Thunder Bay Laboratory

1993

Performance Report

SUMMARY

The Ontario Ministry of Environment and Energy Thunder Bay Laboratory provides analytical support for environmental programs in the Northern Ontario Region. The laboratory performs chemical and microbiological analyses on a wide range of sample types.

The laboratory strives to maintain a high standard of analytical performance through its quality assurance (QA) program, of which quality control (QC) is an important component. The purpose of this report is to summarize the QC data for parameters routinely analyzed in water samples at the Thunder Bay Laboratory. It summarizes types of controls used for a test, the frequency of the controls, and the actual results of controls for the 1993 calendar year. The report is intended as a source of information for the laboratory community and for clients interested in the QC program at the laboratory.

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Titanium
Vanadium
Yttrium
Zinc

1.0 INTRODUCTION

1.1 Quality Control

The purpose of quality control samples is to demonstrate to the operator and data user that the analytical system is in control, and that the data is not affected by the analytical system itself. By monitoring these standards over the long-term, limits of effective performance can be established. By visualizing the performance of the instrument against these limits graphically, deviations from past performance can be easily identified and corrected before data quality is affected.

In addition to using in-house quality control standards, the Thunder Bay Laboratory uses external reference materials, wherever possible, to confirm accuracy and performance of the system. Sources of external reference materials include the National Institute of Standards and Technology (NIST), the National Research Council (NRC) and commercially prepared standard reference materials.

The Thunder Bay Laboratory also actively participates in interlaboratory studies. Interlaboratory studies in which the laboratory regularly participates include:

LRTAP	Long Range Transport of Air Pollutants; Environment Canada; three times per year.
CAEAL	Canadian Association for Environmental Analytical Laboratories; performance evaluation samples, twice per year.
GLAP	Great Lakes Action Plan; Environment Canada; twice per year.
MOEE	Ontario Ministry of Environment and Energy; performance audit samples are sent by the Quality Management Office routinely; special interlaboratory studies are periodically set-up; also referred to as "QM Blind Audits" through-out the report.
MOH	Ontario Ministry of Health; bacteriological samples, twice per year.

Results of interlaboratory studies, or any information regarding this report, can be obtained by contacting C. M. Cotter at (807)475-1763.

1.2   Accreditation and Certification

The laboratory is accredited by the Canadian Association for Environmental Analytical Laboratories (CAEAL/ACLAE Inc.). In order to obtain accreditation by CAEAL, member laboratories must undergo an on-site inspection once every two years and obtain a score ≥70 on performance evaluation (PE) samples; PE samples are sent to laboratories twice per year. Parameters that have the designation of "certified" have obtained a score of ≥70 on PE samples, but have not yet had the opportunity to be evaluated by assessors. Listed below are parameters which are currently accredited by CAEAL; further tests will be accredited as they become available.

Alkalinity - total	BOD
Chloride	Conductivity
Calcium - dissolved	Cadmium - dissolved
Cobalt - dissolved	Chromium - dissolved
Copper - dissolved	Iron - dissolved
Magnesium - dissolved	Manganese - dissolved
Nickel - dissolved	Lead - dissolved
Vanadium - dissolved	Zinc - dissolved
Fluoride	Potassium
Sodium	Nitrate
Nitrate + Nitrite	Silica
Sulphate	Total Kjeldahl Nitrogen
Total Phosphorus	Total Suspended Solids
Fecal Coliforms	Total Coliforms
pH*	

*certified parameter

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2.0   PERFORMANCE REPORT FORMAT

Each parameter's performance report consists of a summary of the test method, followed by a sheet of tabulated data and a plot which summarizes the performance of the test. Although the same general format is used throughout the report, there may be slight differences in the format, reflecting the differences in the type of testing done in each section. Listed below is a glossary of terms which will aid in the interpretation of the summaries. A separate section on quality controls used in microbiology is included in the appropriate section.

Analytical Procedure: Description of the analytical test procedure.

Blank: Sample prepared in the same manner as field samples, but in which the analyte is known to be absent. Tests for background levels of the analyte in the analytical system.

Calibration: The determination of the relationship between analyte concentration and instrument response.

Calibration
Check Solution: Standards prepared separately from the calibration solutions to ensure proper operation of the instrument and verify the current calibration. Also used as a in-run check to determine calibration drift during analysis.

Control Chart: Relates ongoing analytical performance to control limits.

Control Limits: Limits defined statistically or based on protocol requirements which, when exceeded, trigger analyst intervention.

Digested Spikes: Blanks to which a known amount of analyte is added. These samples are taken through the analytical process to monitor analyte recovery. Comparison to control limits determined from historical data establishes the validity of the current analytical run.

Drift: Control samples to check for drift and/or sensitivity change.

Duplicates: Used to determine ability of the analytical system including the operator to repeat an analysis. Establishes the precision that can be expected from the analytical system. Low level duplicate data is used to statistically determine the detection limit of the system.

Interlaboratory Studies:	Studies that involve the exchange of samples amongst laboratories. The results are reported to the referee agency and compared to the median result generated by the participating laboratories. Provides a snapshot comparison to laboratories conducting similar analyses.
Instrumentation:	Type of instrument used to perform the test.
LIS Test Name Code:	Laboratory Information System (LIS) code for analysis requested.
Method Code:	Quality Management Unit Code for the analytical method.
Method Introduced:	Date method was implemented at laboratory.
Modifications:	Describes modifications to the test since the method was introduced, or provides historical information about the test.
Reference Materials:	Purchased samples containing an known amount of analyte. The reference value is established through round-robin testing or through analysis by several independent analytical techniques. Represents an independent test of the accuracy of an analytical method.
Reporting:	The maximum significant figures used to report the result. The calculated W and T values for that parameter. W and T are low level data qualifiers assigned to results that are at or near the detection limit.
Sampling:	Describes the container and preservative (if applicable) that is to be used for sampling and any special instructions required to ensure the integrity of the test.
Sample Preparation:	Sample preparation techniques which must be performed at the laboratory before analysis.
Sample Type/Matrix:	Sample types that can be routed to the workstation.
Section:	Section responsible for test.
Standard Deviation:	An estimate of the spread of repeated measurements about their average value, obtained under specified conditions.
S_w:	Standard deviation based on within-lab repeatability data which is used in setting warning and control limits.

| <T: | A low-level data qualifier used to indicate that the measured value is a trace amount. Data qualified with <T should be interpreted with caution. |

Units: | Unit of measurement in which results are reported.

<W: | A low-level data qualifier which indicates that no measureable responses were observed under the test conditions. The numerical value indicates the smallest amount that could have been measured under routine conditions. W is derived from the standard deviation of duplicates near zero.

Work Station Code: | LIS code for sample routing to the workstation.

3.0   MICROBIOLOGY

3.1   QUALITY CONTROL PROGRAM

In this report, data is presented which summarizes quality control (Q.C.) procedures used in the membrane filtration test and Presence/Absence test. These QC procedures are used to ensure that an analytical test is working properly and that reported results are accurate and reproducible within the limits of normal statistical variation.

MEMBRANE FILTRATION TEST (MF)

Blank Control Filters

Each sample analyzed by the membrane filter test is separated from the previous sample by introducing a control filter at the beginning of each analysis. The control filter is employed in the same manner as those filters used for sample analyses, however, only sterile buffered rinse water is filtered. The control filter is placed on m Endo-LES medium. If there is only one target growing on the control filter and/or a few background colonies, the control filter is considered positive for growth but the next sample result is not deemed to be compromised. However, if excessive contamination is suspected, the next result will not be reported and, if possible, the analyses will be repeated. As well, the result immediately preceeding the control filter is in question, as the bacteria were likely carried over from this sample.

Duplicate Analysis

Duplicate analysis is conducted on ≥5% of the samples. The data are accumulated for each parameter and differences in duplicate results are sorted according to ranges of colony counts per filter. A mean and within-run standard deviation are calculated, where the standard deviation gives a measure of the repeatability of results.

The mean difference within each range is multiplied by 3.267 to give a precision criteria (PRC) or control limit for a particular range. Graphs for each range of colony counts per filter show how 1993 results compare to the calculated control limit, which is based on the previous 12 months of data. Where excessive bias is suspected, corrective action will be taken.

Media Quality Control

The pH of a medium is monitored after sterilization has taken place. The final pH may vary within specified limits from the recommended value. The medium is checked for sterility by incubating random samples of plates at room temperature. Any bacterial growth will require retesting of the medium for sterility. Confirmation of contamination will result in the rejection of the medium. The batch or lot number of a medium is recorded to determine if any changes in quality occur when batch or lot numbers change.

Selective agar media used in the detection and enumeration of indicator bacteria are tested to ensure their proper functioning. A quantitative QC test of agar media for membrane filter tests involves filtering dilute suspensions of positive and negative cultures, or real samples, in duplicate. The duplicate filters are placed, respectively, on agar plates from the new and previous batches of media. If the new batch of medium meets the past performance of the previous medium, the number of colonies on the respective plates should be approximately the same. Medium is retested or rejected if it fails to meet the past performance of the previous medium. In this report, only those results for a target colony or a real sample are tabulated. Results are recorded and statistically analyzed as for duplicate analysis. Also, to ensure a selective medium is allowing for the growth of target organisms, pure cultures are filtered in duplicate and respective filters are placed on selective and non-selective media. The culture should form approximately the same number of colonies on the selective and non-selective plates. Media is retested or rejected if it appears the selective medium is over-inhibitory. Again, the data is recorded and statistically analyzed as for duplicate analysis. This QC testing was being phased-in during 1993 and data is only available for the fecal coliform parameter.

PRESENCE-ABSENCE (P-A) TESTS

Blank Control P-A Bottles

Every 20 samples, a blank control is prepared by pouring 100 mL of sterile distilled water into a P-A bottle and incubating it along with the regular P-A bottles. The P-A blank bottle is incubated for four days and should remain free of any bacterial growth or colour change. Isolation of indicator organisms in more than one P-A blank control test will require rechecking the sterility of the dilution blanks, the P-A medium and the procedure.

Media Quality Control

A number of checks are performed on the P-A broth including: pH, sterility at 20°C and 35°C and growth reaction of Escherichia coli, a fecal streptococcus and Salmonella typhimurium. If the medium is functioning properly, E. coli will produce a strong acid reaction (yellow colour in the medium) and gas reaction. The fecal streptococcus will produce an acid reaction only and S. typhimurium will produce heavy growth only.

3.2   Microbiology Performance Summaries

<center>### *Escherichia* <u>coli</u></center>

IDENTIFICATION:

LIS Test Name Code:	ECMF	Method Introduced: 1983
Work Station Code:	TBMF	Units: Counts/100 mL
Method Code:	E6019A	Section: Microbiology

SAMPLE TYPE/MATRIX: Surface, waste, and drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and/or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC-IG agar and incubated for 23 ± 1 h at $44.5 \pm 0.2°C$. The temperature is gradually elevated by placing 2 plastic jars containing ice (50 mL of water) in the plastic container (one at each end of the container). The maximum number of target colonies per filter for counting purposes is 100.

CONTROLS AND QUALITY ASSURANCE:

Controls:	Blank filter between each sample.
	Duplicate samples on $\geq 5\%$ of samples.
Media QC:	Comparison of counts of a positive culture and a negative culture on present vs. previous media.
Reporting:	Results are qualified by "A" (for approximate) if the number of target colonies/filter is <10 and ≤ 10 mL of sample was filtered, or if the total number of colonies on a plate >300.

If the number of target organisms exceeds the upper counting limit of 100, the result is reported as: $>\dfrac{(100 \times 100)}{\text{mL filtered.}}$

If there is no recovery of target organisms, the result is reported as: $<\dfrac{(1 \times 100)}{\text{mL filtered.}}$

Escherichia coli

CONTROLS AND QUALITY ASSURANCE (Cont'd):

External QC   m TEC-Ig agar was tested with an E. coli quality control culture (lot
Checks: #121589) from the USEPA.

                     Participation in Ontario Ministry of Health interlaboratory studies.

MODIFICATIONS:

Before 1983, E. coli was detected using a 2-step urease procedure.

In 1993, a study was done to compare methods for the recovery of E. coli. Based on the results
of the study, all Ontario Ministry of Environment and Energy and Ontario Ministry of Health
Laboratories are using m FC-BCIG medium for the recovery of E. coli as of 1994.

Escherichia coli — ECMF

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 — 100 Counts per Filter

Controls:

Number of Controls	Positive Controls
324	0

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
50	0—20	2	1.4
20	21—80	5	3.3
0	81—100	—	—

Media Quality Control:

Comparison of E. coli on Present vs. Previous Medium:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
0	0—20	—	—
9	21—80	2.6	2.5
2	81—100	5	—

Escherichia coli
0–20 Counts per Filter

Absolute Difference Between Pairs

Pair No. — Control Limit
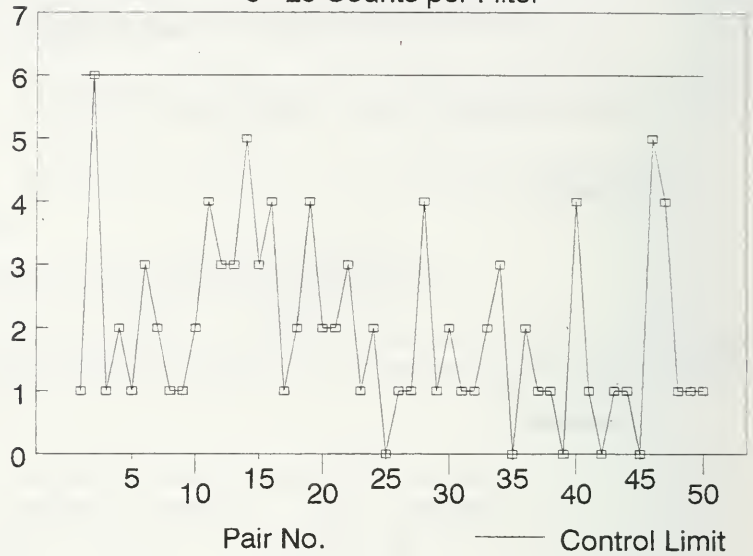
Escherichia coli
21–80 Counts per Filter

Absolute Difference Between Pairs

Pair No. — Control Limit

14
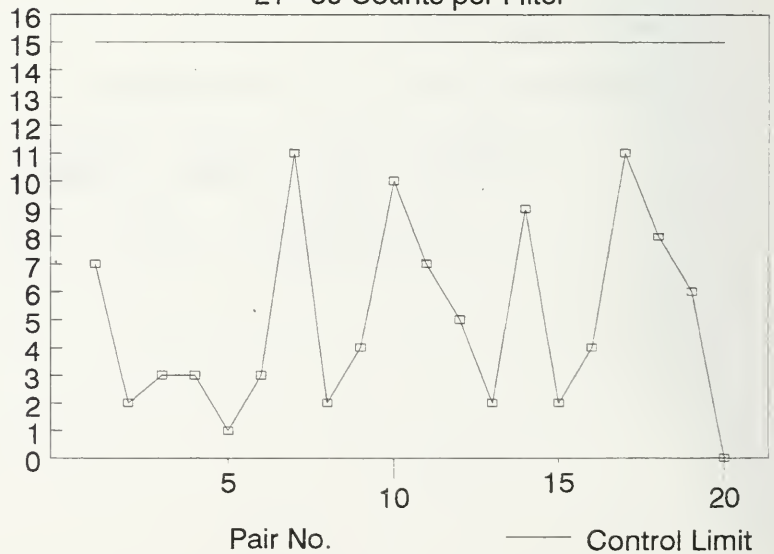
FECAL COLIFORMS

IDENTIFICATION:

LIS Test Name Code:	FCMF	Method Introduced: 1978
Work Station Code:	TBMF	Units: Counts/100 mL
Method Code:	E6018A	Section: Microbiology

SAMPLE TYPE/MATRIX: Surface, waste, and drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and/or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mTEC agar and incubated for 23 $\pm$ 1 h at $44.5 \pm 0.2°C$. The temperature is gradually elevated by placing 2 plastic jars containing ice (50 mL of water) into the plastic container (one at each end of the container). The maximum number of target colonies per filter for counting purposes is 100.

CONTROLS AND QUALITY ASSURANCE:

Controls:	Blank filter between each sample.
	Duplicate samples on $\geq$ 5% of samples.
Media QC:	Target organism count on selective vs. non-selective media.
	Comparison of a real sample on present vs. previous media.
Reporting:	Results are qualified by "A" (for approximate) if the number of target colonies/filter <10 and $\leq$10 mL of sample was filtered, or if the total number of colonies is >300.

If the number of target organisms exceeds the upper counting limit of 100, the result is reported as: $>\dfrac{(100 \times 100)}{\text{mL filtered.}}$

FECAL COLIFORMS

CONTROLS AND QUALITY ASSURANCE (Cont'd):

Reporting: If there is no recovery of target organisms, the result is reported as:
$$\frac{<(1 \times 100)}{mL\ filtered.}$$

External QC   CAEAL accredited.
Checks:

mTEC agar was tested with an E. coli quality control culture (lot #121589) from the USEPA.

Participation in Ontario Ministry of Health Interlaboratory studies.

MODIFICATIONS:

Before 1978, fecal coliforms were detected using m FC medium.

Fecal Coliforms — FCMF

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 — 100 Counts per Filter

Controls:

Number of Controls	Positive Controls
1873	0

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
166	0−20	1.6	1.6
18	21−80	5.2	2.9
0	81−100	−	−

Media Quality Control:

Present vs Previous Medium Using a Real Sample:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
6	0−20	0.7	0.7
5	21−80	7.2	4.7
0	81−100	−	−

E. coli on Selective vs Non−selective Medium:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
1	0−20	7	−
9	21−80	4.6	5.1
1	81−100	13	−

Fecal Coliforms
0–20 Counts per Filter
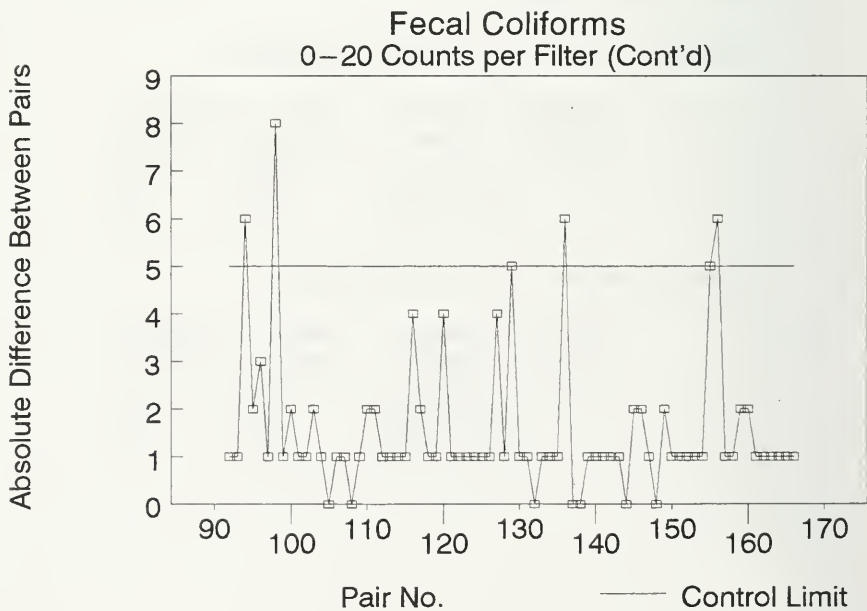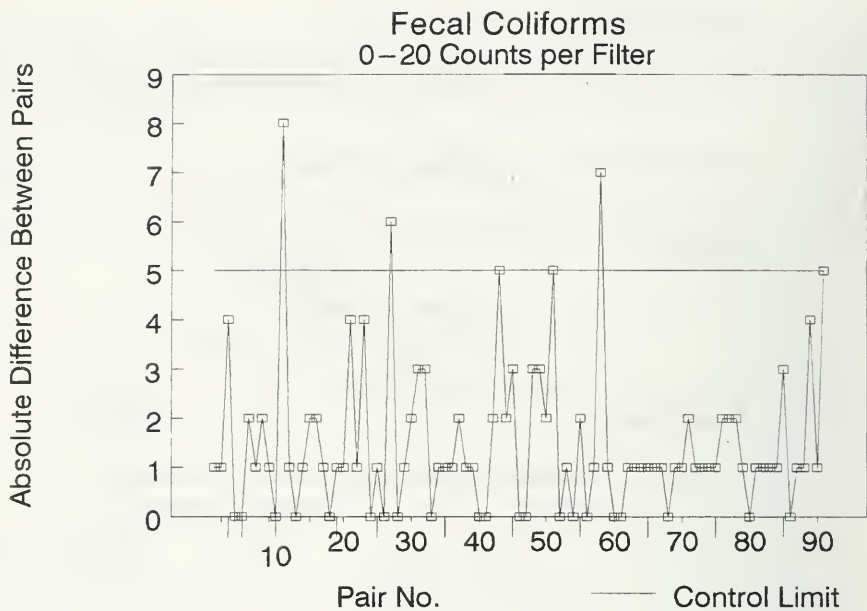
Fecal Coliforms
0–20 Counts per Filter (Cont'd)

Fecal Coliforms
21−80 Counts per Filter
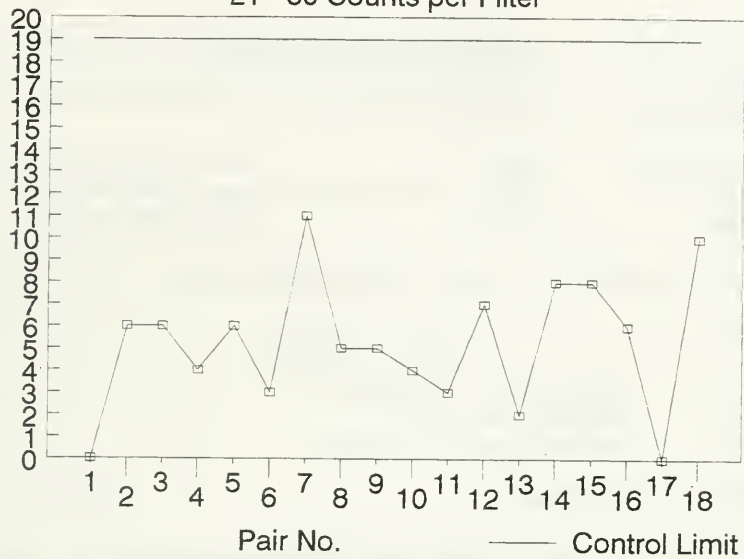
FECAL STREPTOCOCCUS

IDENTIFICATION:

LIS Test Name Code:	FSMF	Method Introduced: 1968
Work Station Code:	TBMF	Units: Counts/100 mL
Method Code:	E6017A	Section: Microbiology

SAMPLE TYPE/MATRIX: Surface, waste, and drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and/or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the the surface of mEnterococcus agar and incubated for 48 ± 2 hours at 35 ± 0.5°C. All colonies that are red, maroon or pink are counted as fecal streptococcus. The maximum number of target colonies per filter for counting purposes is 150.

CONTROLS AND QUALITY ASSURANCE:

Controls:	Blank filter between each sample. Duplicate samples on $\geq$ 5% of samples.
Media QC:	Comparison of counts of a positive culture and a negative culture on present vs. previous media.
Reporting:	Results are qualified by "A" (for approximate) if the number of target colonies/filter is <10 and ≤10 mL of sample was filtered, or if the total number of colonies on a plate >300.

If the target colony count/filter exceeds the upper counting limit of 150, the result is reported as $>\dfrac{(150 \times 100)}{\text{mL filtered.}}$

If there is no recovery of target organisms, the result is recorded as $<\dfrac{(1 \times 100)}{\text{mL filtered.}}$

Fecal Streptococcus — FSMF

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 — 150 Counts per Filter

Controls:

Number of Controls	Positive Controls
1331	0

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
168	0 – 20	1.5	1.2
20	21 – 80	5.4	4.6
0	81 – 150	–	–

Media Quality Control:

Fecal Streptococcus on Present vs. Previous Medium:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
9	0 – 20	2.4	2.1
4	21 – 80	3.5	2.1
0	81 – 150	–	–

Fecal Streptococcus
0–20 Counts per Filter

Fecal Streptococcus
0–20 Counts per Filter (Cont'd)

22
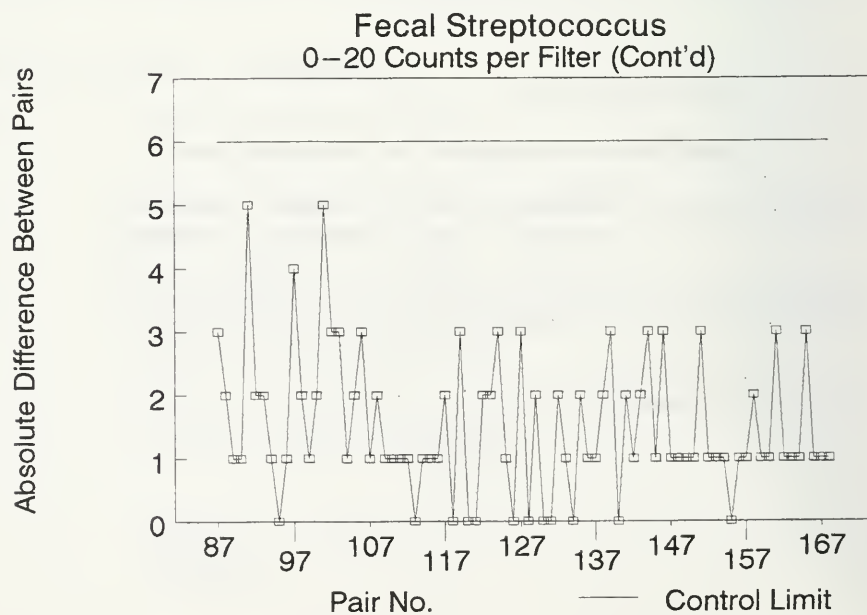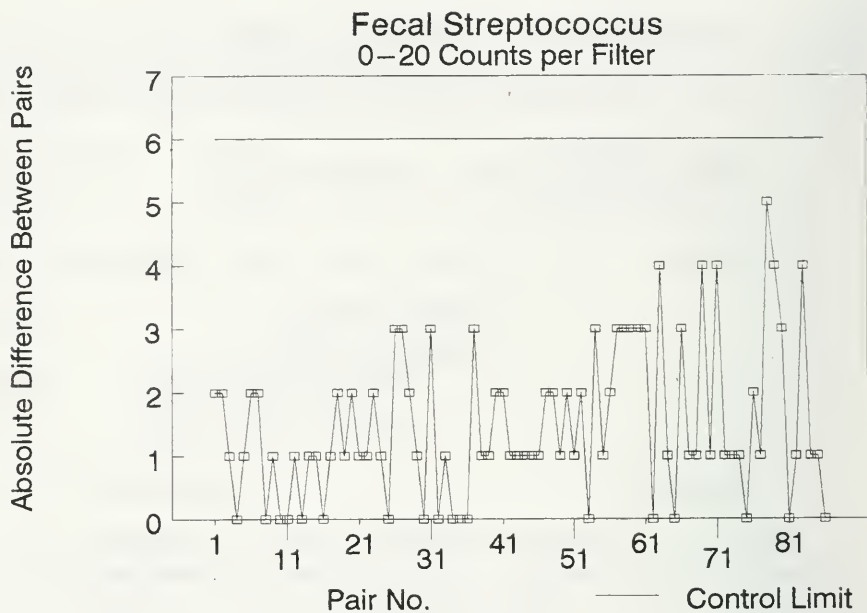
Fecal Streptococcus
21–30 Counts per Filter

Absolute Difference Between Pairs

Pair No.   Control Limit
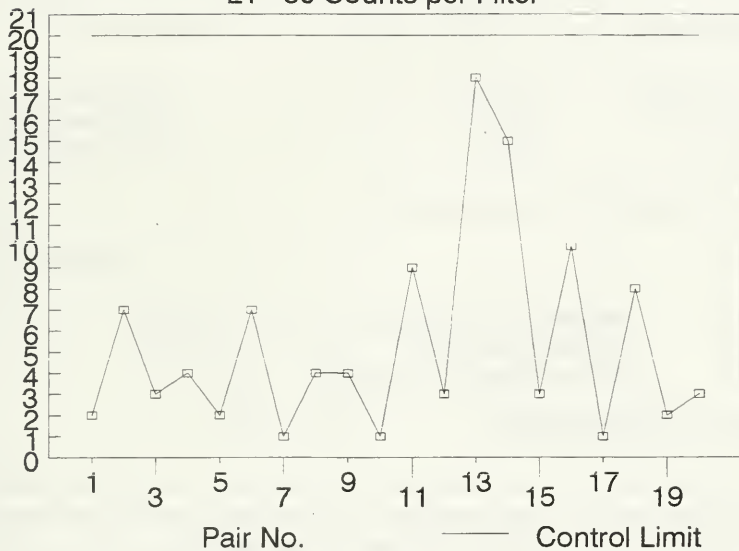
HETEROTROPHS

IDENTIFICATION:

LIS Test Name Code:	HB35MF	Method Introduced: 1979
Work Station Code:	TBMF	Units: Counts/mL
Method Code:	E6000A	Section: Microbiology

SAMPLE TYPE/MATRIX: Drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample is filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of SPC agar and incubated for
48 ± 3 hours at $35 \pm 0.5°C$. All colonies are counted as heterotrophs.

CONTROLS AND QUALITY ASSURANCE:

Controls: Duplicate samples on $\geq 5\%$ of samples.

Media QC: Comparison of colony counts on present vs. previous media.

Reporting: Results are qualified by "A" (for approximate) if the number of colonies >1000.

External QC Checks: SPC agar was tested with an E. coli quality control culture (lot #121589) from the USEPA.

Heterotrophic Plate Count — HPC

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 — 1000 Counts per Filter

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
177	0−20	1.9	1.9
29	21−80	7.2	4.9
14	81−300	11.4	7.4

Media Quality Control:

E. coli on Present vs. Previous Medium:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
3	0−20	1	1.4
15	21−80	4.7	3.3
1	81−300	10	−

Heterotrophic Plate Count
0–20 Counts per Filter

Absolute Difference Between Pairs

Pair No.   ——— Control Limit

Heterotrophic Plate Count
0–20 Counts per Filter (Cont'd)

Absolute Difference Between Pairs

Pair No.   ——— Control Limit

26
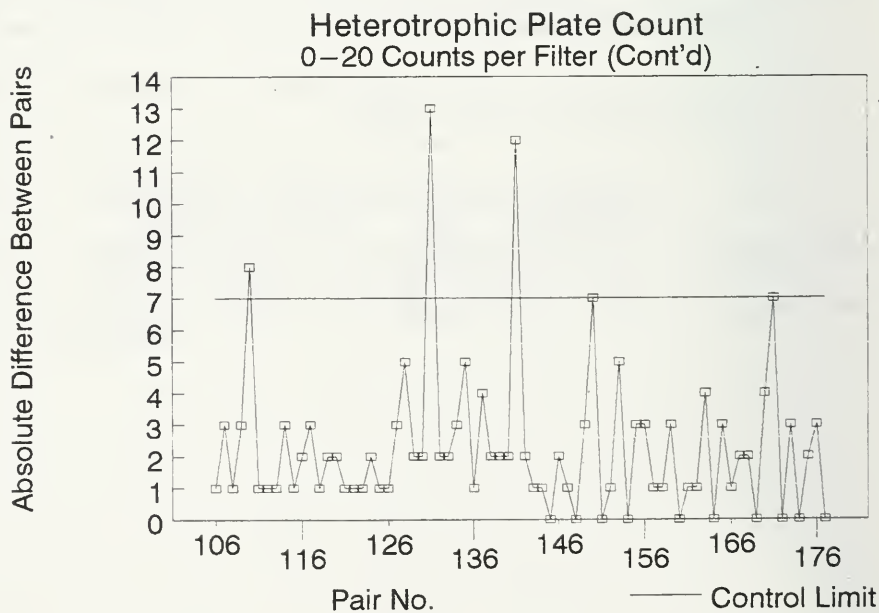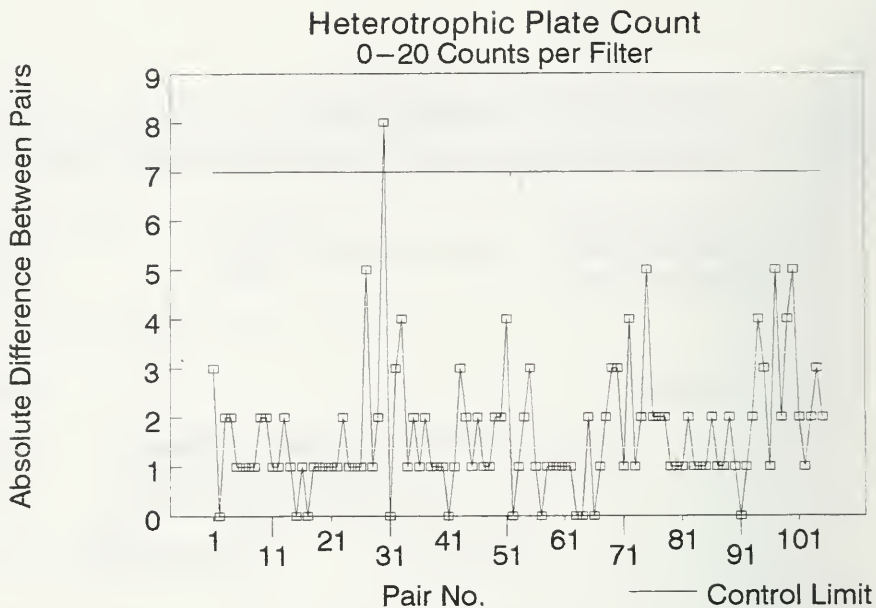
Heterotrophic Plate Count
21–80 Counts per Filter
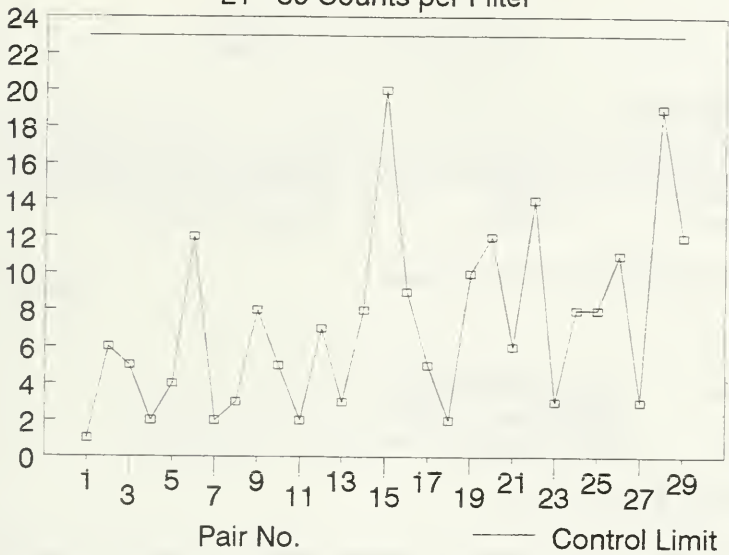
Heterotrophic Plate Count
>81 Counts per Filter

27
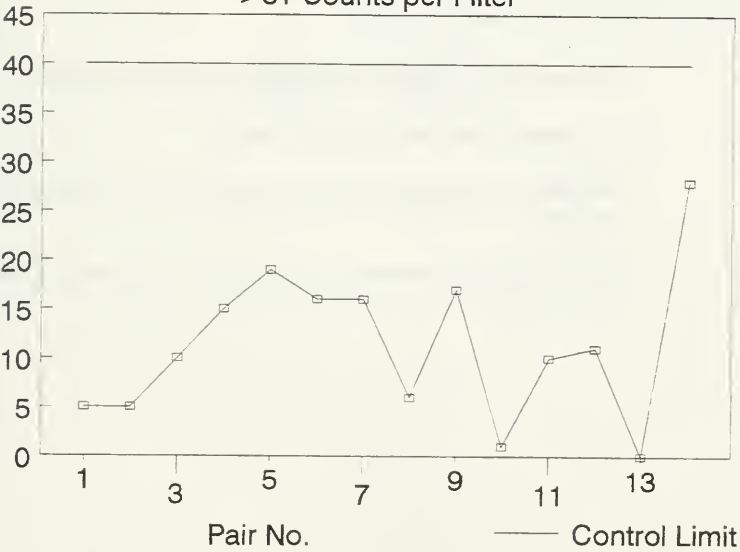
PRESENCE-ABSENCE TEST

IDENTIFICATION:

LIS Test Name Code:	TBPA	Method Introduced: 1969
Work Station Code:	TBPA	Units: Present/Absent/100 mL
Method Code:	E6022A	Section: Microbiology

SAMPLE TYPE/MATRIX: Drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

A 100 mL volume of sample is added to a presence-absence (P-A) bottle. The bottle is incubated at $35 \pm 0.5°C$ for 4 days and examined every 24 hours for acid and/or gas formation. When a positive reaction occurs, the inoculum is transferred to confirmatory media to determine the presence of total coliforms, fecal coliforms, E. coli and other indicator organisms.

CONTROLS AND QUALITY ASSURANCE:

Controls:	A blank control sample is included every 20 to 25 samples.
Media QC:	PA broth batches are checked for sterility.
	Inoculation of the medium is done with various organisms to determine its response.
Reporting:	Microbiological parameters are reported as either present or absent per 100 mL of sample.

Presence—Absence — TBPA

Quality Control Data from January 1 to December 31, 1993

Controls:

		Controls with:	
Number of Samples	Number of Controls	growth	indicator organisms
14322	787	10	0

Media Quality Control:

Number of Batches	Batches Rejected
20	0

Pseudomonas aeruginosa

IDENTIFICATION:

LIS Test Name Code:	PSAMF	Method Introduced: 1978	
Work Station Code:	TBMF	Units: Counts/100 mL	
Method Code:	E6016A	Section: Microbiology	

SAMPLE TYPE/MATRIX: Surface, waste and drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and/or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mPAE agar and incubated for 48 ± 2 hours at 41.5 ± 0.5°C. All colonies that are dark brown/brown with darkened centers are counted as Pseudomonas aeruginosa. The maximum target colonies counted per plate is 150.

CONTROLS AND QUALITY ASSURANCE:

Controls: Blank filter between each sample
Duplicate samples on ≥ 5% of samples

Media QC: Comparison of counts of a positive culture and a negative culture on present vs. previous media.

Reporting: Results are qualified by "A" (for approximate) if the number of target colonies/filter is <10 and ≤10 mL of sample was filtered, or if the total number of colonies on a plate is >300.

If the target colony count/filter exceeds the upper counting limit of 150, the result is reported as: $>\dfrac{(150 \times 100)}{mL\ filtered.}$

If there is no recovery of target organism, the result is recorded as: $<\dfrac{(1 \times 100)}{mL\ filtered.}$

MODIFICATIONS:

Medium was changed from m PAE to m PAC on October 8, 1993.

Pseudomonas aeruginosa – PSAMF

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 – 150 Counts per Filter

**Controls:**

Number of Controls	Positive Controls
228	0

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
22	0 – 20	2	1.4
4	21 – 80	2.8	2.3
0	81 – 150	–	–

Media Quality Control:

Pseudomonas aeruginosa on Present vs. Previous Medium:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
4	0 – 20	2.8	0.8
3	21 – 80	1	1.4
0	81 – 150	–	–

Pseudomonas aeruginosa
0–20 Counts per Filter

Absolute Difference Between Pairs vs. Pair No.

Control Limit
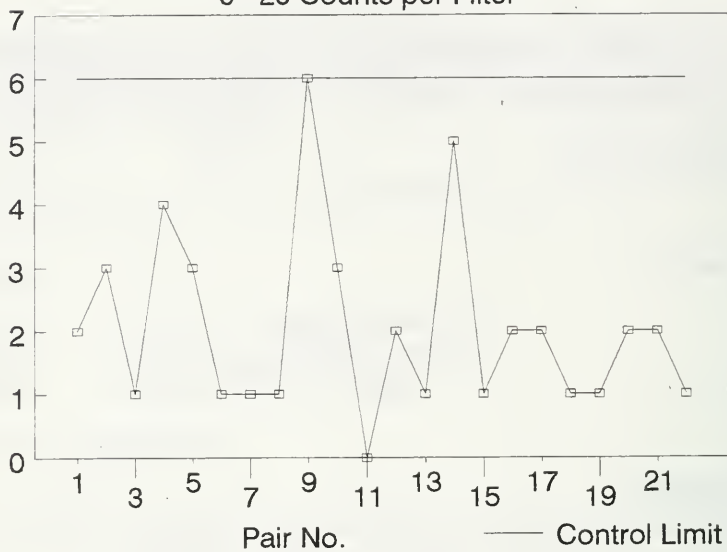
TOTAL COLIFORMS

IDENTIFICATION:

LIS Test Name Code:	TCMF	Method Introduced: 1968	
Work Station Code:	TBMF	Units: Counts/100 mL	
Method Code:	E6023A	Section: Microbiology	

SAMPLE TYPE/MATRIX: Surface and drinking water.

SAMPLING:

Requirements:	Bottle filled to top of label
Container:	250 mL glass or plastic
Preservative:	Sodium thiosulfate, 100 mg/L

ANALYTICAL PROCEDURE:

Samples are analyzed by the membrane filter (MF) procedure using aseptic technique. The sample and/or dilution water are filtered through a water permeable membrane which traps the bacteria on the filter. The filter is placed onto the surface of mENDO-LES agar and incubated for 22 ± 2 hours at $35 \pm 0.5°C$. All colonies with a dull to bright metallic green-gold sheen are counted as coliforms. The maximum number of target colonies per filter for counting purposes is 150.

CONTROLS AND QUALITY ASSURANCE:

Controls: Blank control filter between each sample.
Duplicate samples on $\geq 5\%$ of samples.

Media QC: Target and non-target organisms are filtered onto mENDO-LES.

Reporting: Results are qualified by "A" (for approximate) if the number of target colonies/filter is <10 and ≤10 mL of sample was filtered, or if the total number of colonies on a plate >300.

If the target colony count/filter exceeds the upper counting limit of 150, the result is reported as $>\dfrac{(150 \times 100)}{\text{mL filtered.}}$

If there is no recovery of target organisms, the result is recorded as $<\dfrac{(1 \times 100)}{\text{mL filtered.}}$

33

TOTAL COLIFORMS

CONTROLS AND QUALITY ASSURANCE (Cont'd)

External QC   CAEAL accredited.
Checks:
             mENDO-LES agar was tested with an E. coli quality control culture (lot #121589) from the USEPA.

             Participation in Ontario Ministry of Health Interlaboratory studies.

Total Coliforms — TCMF

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0 — 150 Counts per Filter

Controls:

Number of Controls	Positive Controls
2644	0

Duplicates:

Number of Data Pairs	Counts per filter	Mean Difference	Standard Deviation
210	0 – 20	1.4	1.3
24	21 – 80	4	2.6
5	81 – 150	9.6	5.6

Media Quality Control:

Batches Prepared	Batches Rejected
53	0

Total Coliforms
0−20 Counts per Filter

Total Coliforms
0−20 Counts per Filter (Cont'd)

Total Coliforms
21–80 Counts per Filter

Absolute Difference Between Pairs

Pair No.   ——— Control Limit
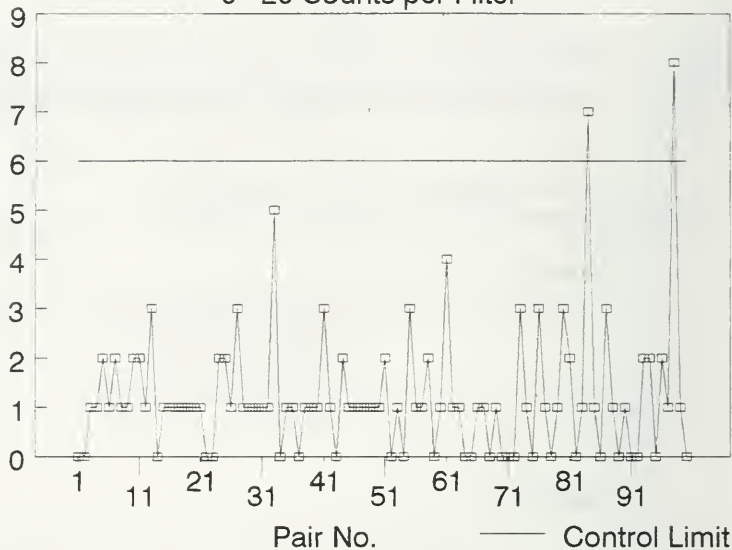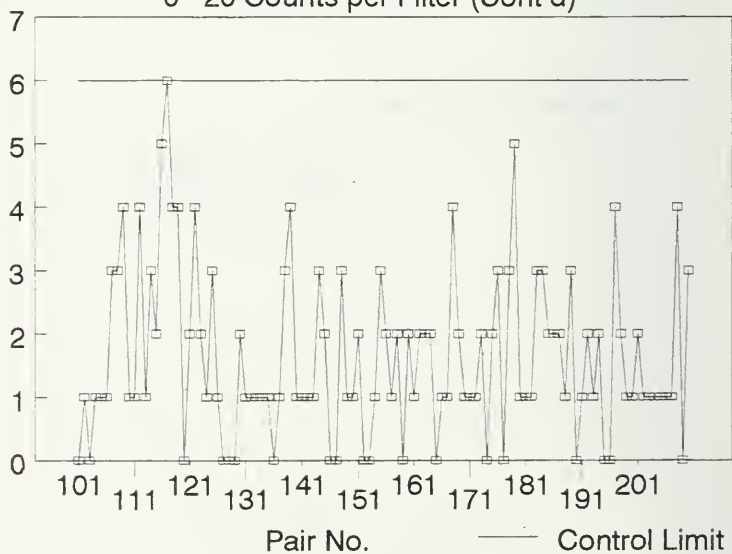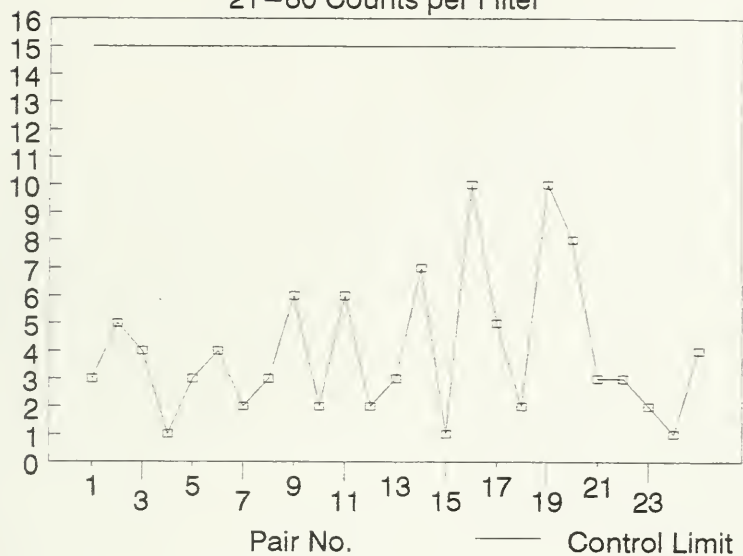
4.0   Water Quality Performance Summaries

ACIDITY - TOTAL FIXED ENDPOINT

IDENTIFICATION:

LIS Test Name Code:	ACDT	Introduced:	78/08/01
Work Station Code:	TBACDT	Units:	mg/L as $CaCO_3$
Method Code:	E6001A.1	Section:	Water Quality

SAMPLE TYPE/MATRIX: Surface water, drinking water, ground water, snow, precipitation and industrial waste.

SAMPLING:

Special Instructions:	Bottles to be completely full so no air remains after capping.
Container:	PET or glass
Preservative:	4°C

ANALYTICAL PROCEDURE:

Sample aliquot (50 mL) is titrated with CO_2 free standardized 0.02N NaOH to a pale pink colour (endpoint pH 8.3), using phenolphthalein as the indicator. A pH meter is used to distinguish the endpoint in highly coloured samples.

INSTRUMENTATION:

Brinkman Autoburette with digital display.
PH meter accurate to 2 decimal places.

CALIBRATION:

Potassium Hydrogen Phthalate titrated against the NaOH working standard.

CONTROLS AND QUALITY ASSURANCE:

Duplicates: DUP (1 every 10 samples, minimum - 1 per run)

Reporting: Maximum Significant Figures: Whole Numbers
              W Value: N/A   T Value: N/A

MODIFICATIONS:

1986-Phenolphthalein indicator used for end-point determination. pH meter used exclusively prior to this date.

ACIDITY

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 40 mg/L as CaCO3

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
39	0 – 8	4.051	0.3922
8	8 – 20	11.625	0.4330
1	20 – 40	23.000	0.7071

ALKALINITY - GRAN

IDENTIFICATION:

LIS Test Name Code:	ALKTI	Introduced:	1980
Work Station Code:	TBTFE	Units:	mg/L CaCO3
Method Code:	E6010A	Section:	Water Quality

SAMPLE TYPE/MATRIX: Surface water and precipitation samples with suspected low alkalinities.

SAMPLING:

Special Instructions:	Fill bottle completely.
Container:	PET or glass
Preservative:	4°C

ANALYTICAL PROCEDURE:
An aliquot of sample (usually 100 mLs) is titrated with 0.02N sulphuric acid under the control of a microcomputer to a pH end-point of <3.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the Ph reading following each aliquot of titrant. Once a complete titration curve is obtained, the Gran method of analysis is used to determine the end-point volume and this, in turn, is used to calculate the Inflection Point (IP) alkalinity.

INSTRUMENTATION:
Auto-Burette, Radiometer ABU91,with 5.0 mL total delivery burette assembly and BCD internal pH meter, combination pH electrode, microcomputer with appropriate software (in-house design).

CALIBRATION: 3 standard buffers - pH value 4.01, 6.86 and 9.18.

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA AND QCB
Duplicates:	DUP (1 for every 15 samples, run at beginning)

Reporting:	Maximum Significant Figures: 3
	W Value: N/A T Value: N/A

LRTAP participant

MODIFICATIONS:

Sept. 1992 - TRS-80 computer and software was replaced by Radiometer Auto-Burette and microcomputer with in-house software.

NOTE: New instrumentation, insufficient data for charting.

ALKALINITY - TOTAL FIXED ENDPOINT

IDENTIFICATION:

LIS Test Name Code:	ALKT	Introduced:	1978
Work Station Code :	TBCAP	Units:	mg/L CaCO3
Method Code:	E6003A	Section:	Water Quality

SAMPLE TYPE/MATRIX:
Surface water, drinking water, groundwater, industrial waste and sewage effluents.

SAMPLING:

Special Instructions:	Fill bottle completely.
Container:	PET or glass
Preservative:	4°C

ANALYTICAL PROCEDURE:
Samples are titrated with 0.02N sulphuric acid to pH <4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

INSTRUMENTATION:
Radiometer CDM 83 Conductivity Meter, TTT85 titrator, ABU80 Auto-Burette, PRS12 Alpha Printer and an SAC80 Multisampler.

CALIBRATION: 3 standard buffers - pH value 4.01, 6.86 and 9.18.

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA AND QCB
Duplicates:	DUP (1 for every 15 samples, run at beginning)

Reporting:	Maximum Significant Figures: 3
	W Value: N/A T Value: N/A

CAEAL Accredited, LRTAP and QM Blind Audit participant.

MODIFICATIONS:

1986-Fisher Titralizer system replaced by Radiometer system.
1990-Long Term Blank now subtracted from Quality Control Solutions before charting.

ALKALINITY – TOTAL FIXED ENDPOINT

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 200.0 mg/L as CaCO3

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	179	50	49.793	−0.207	0.2855
QCB:	179	25	24.854	−0.146	0.2062
QCC:	87	5	4.885	−0.115	0.0860
QCA+QCB:	179	75	74.648	−0.352	0.4290
QCA−QCB:	179	25	24.939	−0.061	0.2530
QCB+QCC:	87	30	29.723	−0.277	0.2343
QCB−QCC:	87	20	19.954	−0.046	0.1483

For 1993 Control Charts:

$$Sw (A-B) = \quad 0.2763$$
$$Sw(B-C) = \quad 0.2763$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
189	0	− 40	18.499	0.2644
172	40	− 100	63.087	0.4843
95	100	− 200	141.945	0.5721

Alkalinity-Total, QCA

QCA
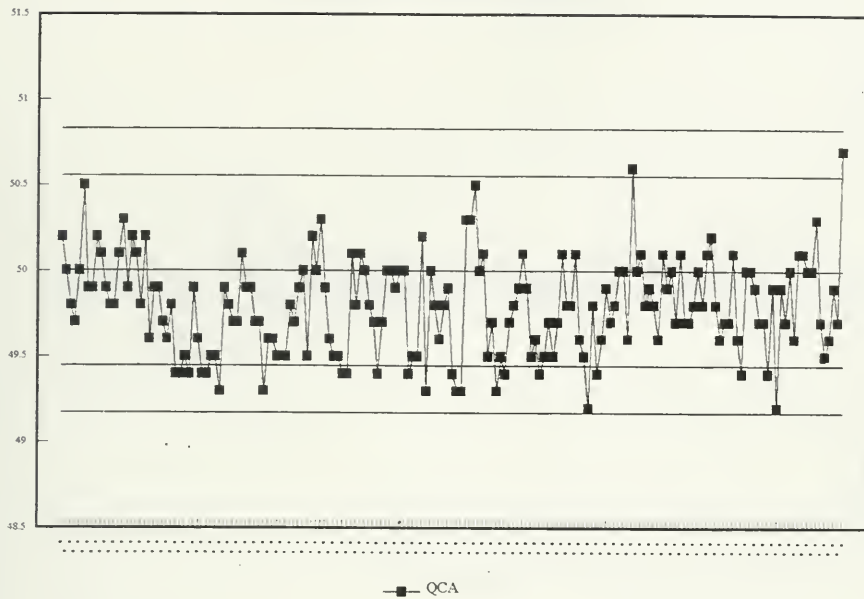
Alkalinity-Total, QCB

QCB
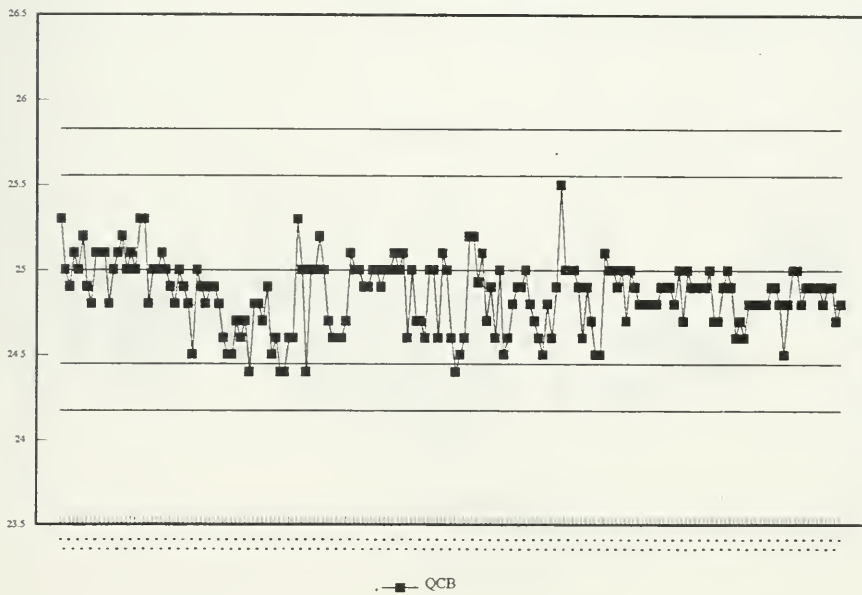
Alkalinity-Total, QCC

QCC
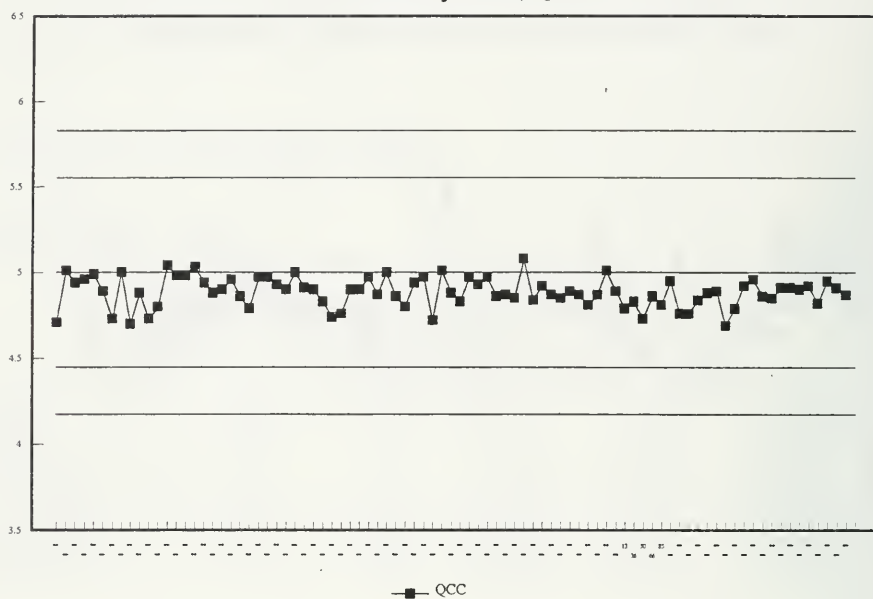
Alkalinity-Total, QC Sum

QCA+QCB

46
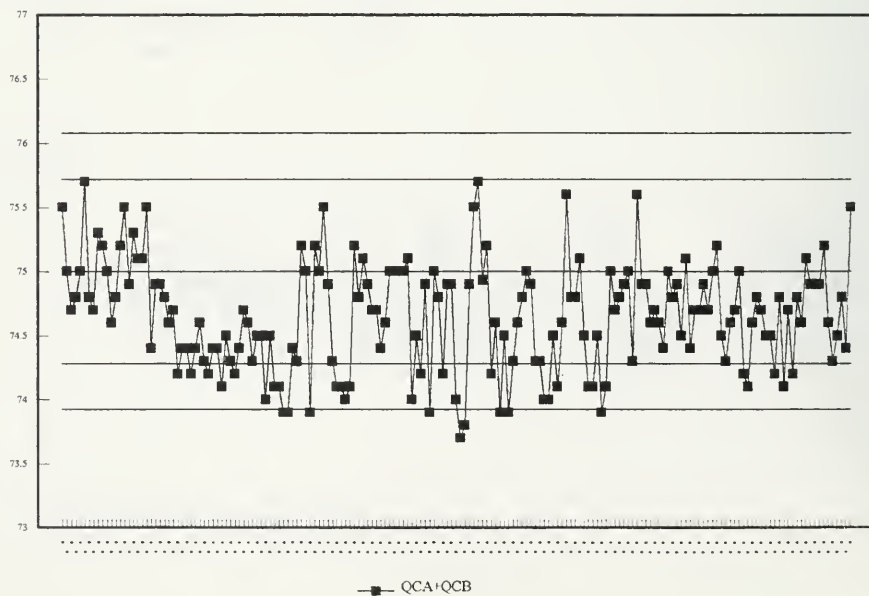
Alkalinity-Total, QC Difference

QCA-QCB
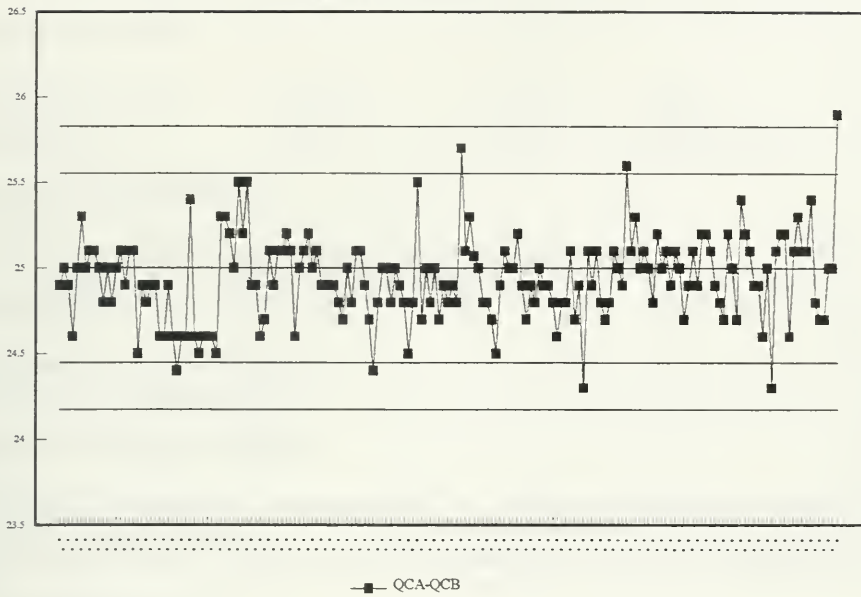
Alkalinity-Total, QCC Sum

QCB+QCC

47
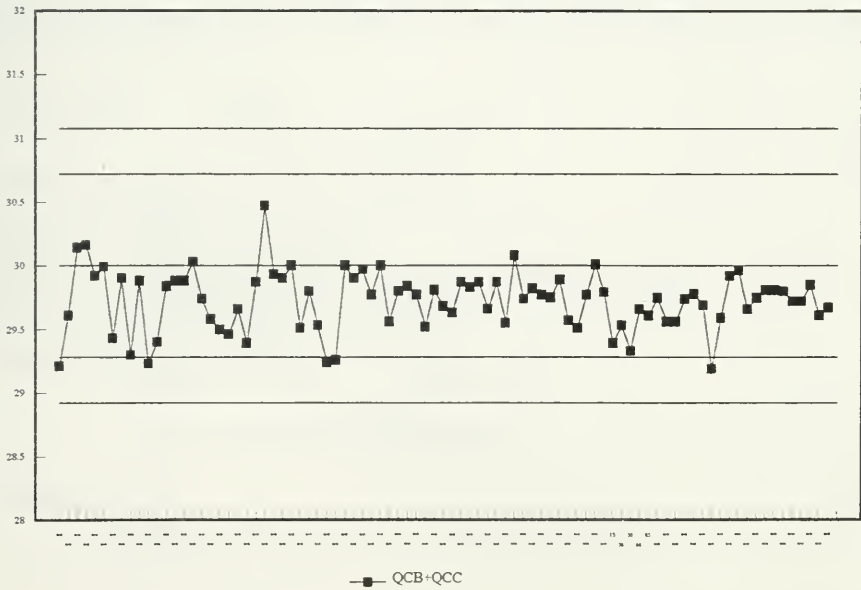
Alkalinity-Total, QCC Difference

QCB-QCC

48
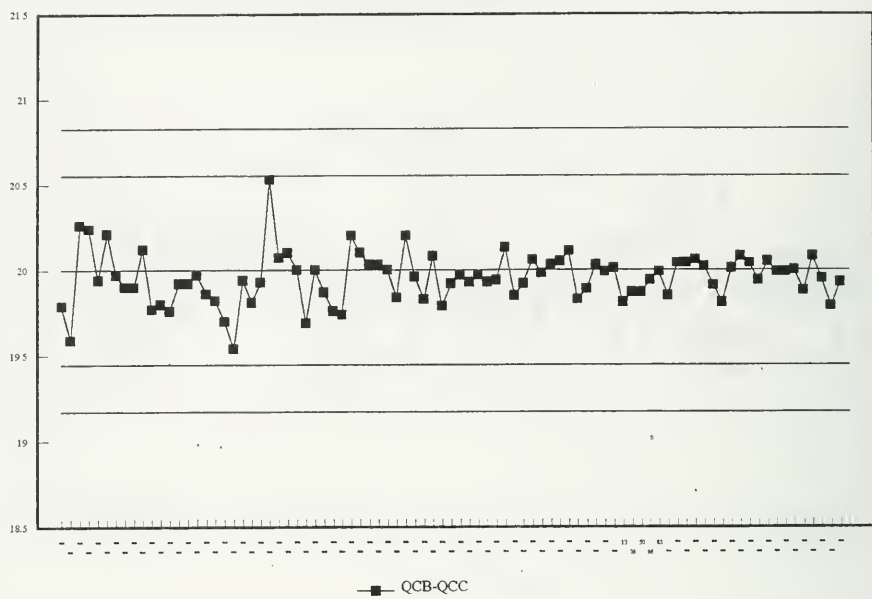
AMMONIA

IDENTIFICATION:

LIS Test Name Code:	NNHTFR	Introduced:	1978
Work Station Code :	TBNDNP	Units:	mg/L as N
Method Code:	E6024A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Ammonia is converted to indophenol blue in a buffered alkaline media using sodium nitroprusside as a catalyst.
N.B. Nitrate plus nitrite, nitrite and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Automated continuous flow system, Technicon AAII with 37°C heating bath. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION: - Linear
                    - 7 Standards   0 - 0.80 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: BLK every 10 samples, CHK (100%) every 20 samples
Duplicates: DUP (3 per run, run at beginning)

Reporting: Maximum Significant Figures: 3
              W Value: 0.01   T Value: 0.05

              LRTAP and QM Blind Audit participant.

MODIFICATIONS:

1988 - All channels went to microcomputer control with DCI software.

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 0.80 mg/L as N

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	84	0.640	0.636	−0.004	0.0060
QCB:	84	0.160	0.156	−0.004	0.0026
QCC:	84	0.064	0.060	−0.004	0.0021
QCA+QCB:	84	0.800	0.792	−0.008	0.0071
QCA−QCB:	84	0.480	0.480	0.000	0.0060
QCB+QCC:	84	0.224	0.216	−0.008	0.0039
QCB−QCC:	84	0.096	0.095	−0.001	0.0026

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.0054$$
$$Sw\ (B-C) = \quad 0.0045$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
188	0.00 − 0.16	0.028	0.0038
30	0.16 − 0.40	0.263	0.0074
11	0.40 − 0.80	0.550	0.0097

Ammonia, QCA

Ammonia, QCB

51

Ammonia, QCC

QCC

Ammonia, QC Sum

QCA+QCB

52
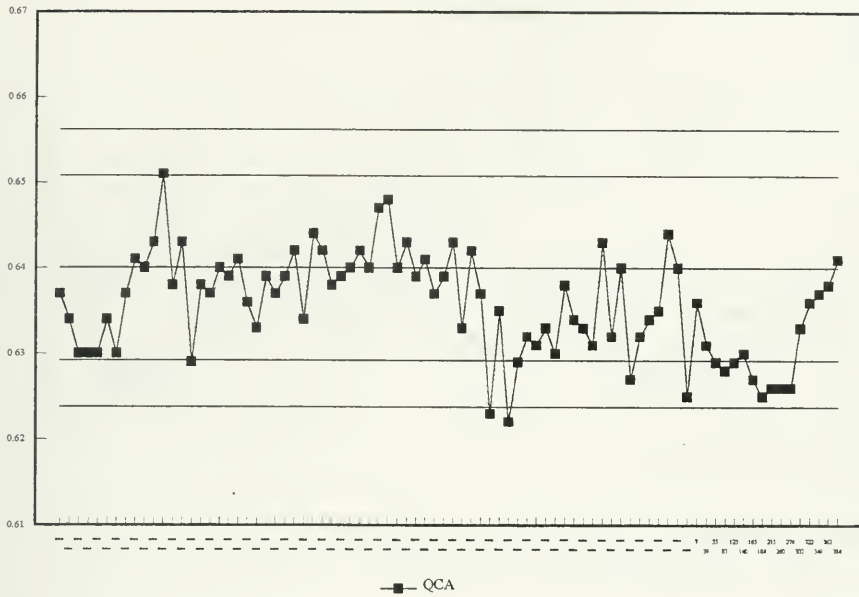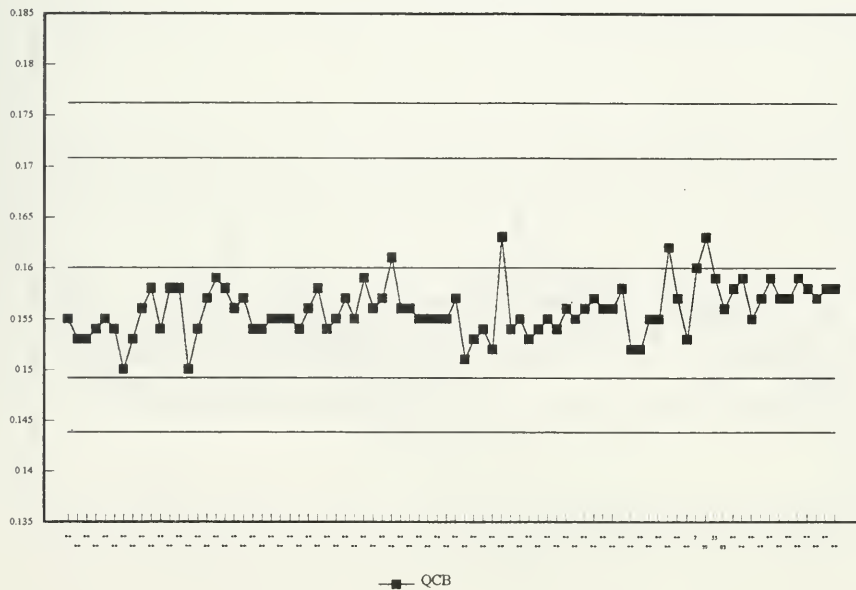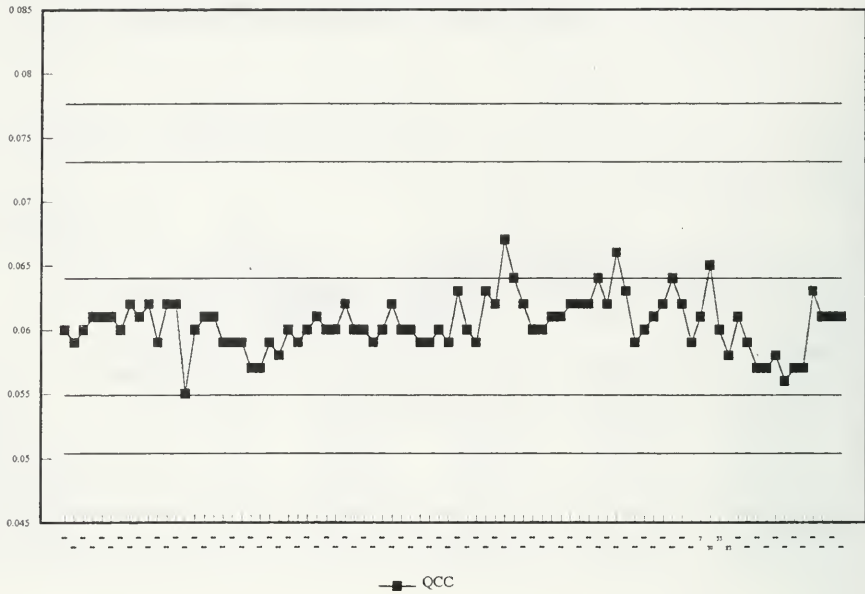
Ammonia, QC Difference

QCA-QCB

Ammonia, QCC Sum

QCB+QCC

53
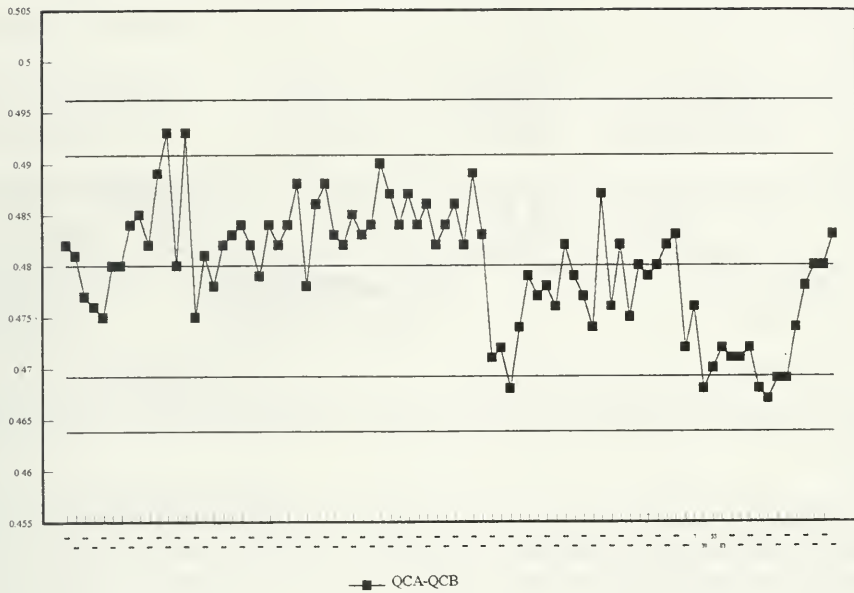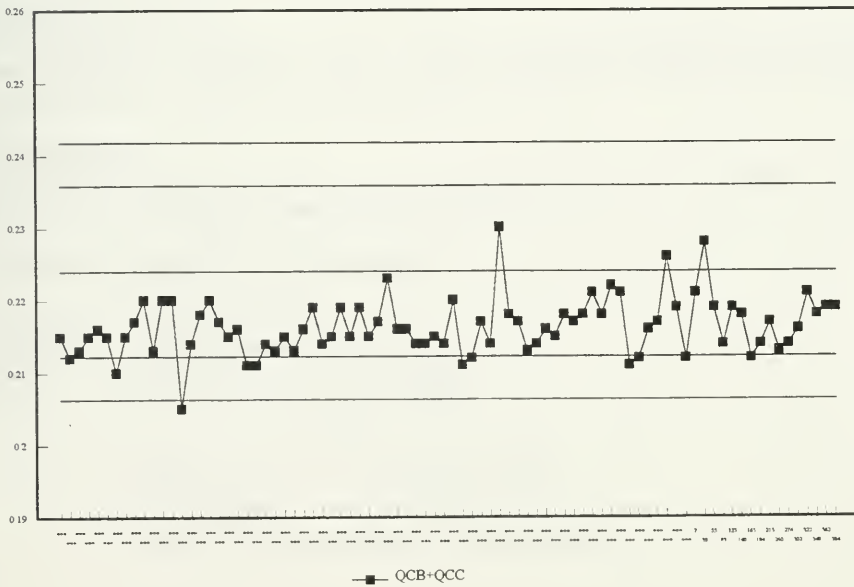
Ammonia, QCC Difference
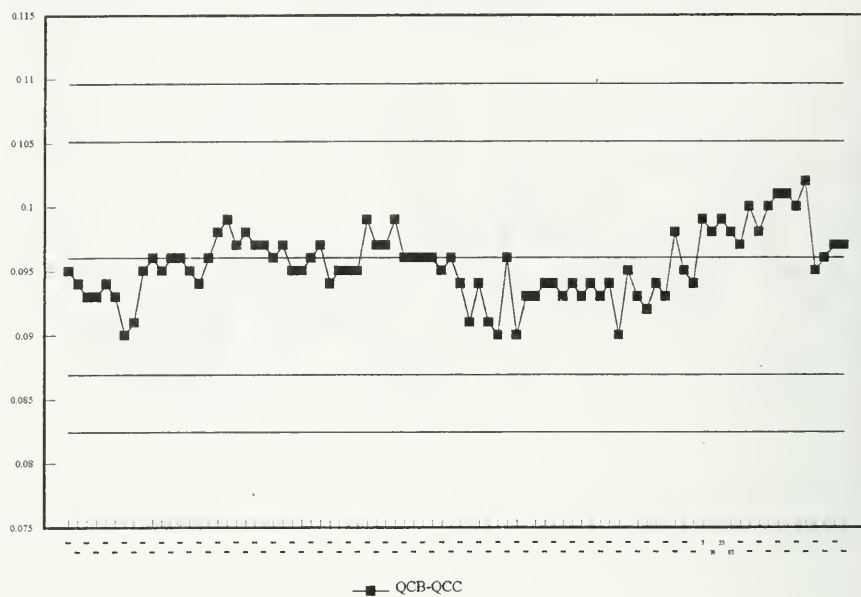
BIOCHEMICAL OXYGEN DEMAND

IDENTIFICATION:

LIS Test Name Code:	BOD5	Introduced:	May, 1981
Work Station Code :	TBBOD5	Units:	mg/L as BOD
Method Code:	E6027A	Section:	Water Quality

SAMPLE TYPE/MATRIX: Sewages, industrial wastes, landfill leachates and surface waters.

SAMPLING:
Special Instructions: Store in dark.
Container: PET or glass
Preservative: Refrigerate at 4°C.

SAMPLE PREPARATION:
If necessary, sample pH is adjusted to neutral and chlorine is removed by reaction with sodium thiosulphate.

ANALYTICAL PROCEDURE:
Using dissolved oxygen (DO) analysis, samples are measured for oxygen depletion before and after a five day period of storage in the dark at 20°C. Dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone a preparation step as above, has been frozen or is an industrial waste, a sewage seed is added and the appropriate seed correction is made.

INSTRUMENTATION:
YSI Oxygen Meter, Model 58, and YSI DO probe with stirrer fitted with a high sensitivity membrane which is permeable to oxygen. Titration equipment for Winkler analysis of dissolved oxygen. Incubator capable of maintaining 20°C ± 1°C.

CALIBRATION:
Dilution water is analyzed by Winkler titration for DO concentration. This water, now with a "known" DO value is used to standardize the oxygen meter.

CONTROLS AND QUALITY ASSURANCE:

Calibration:	2 DO samples, QCA, QCB
Recovery:	3 Recovery Samples
Drift:	DO sample after every six samples
Duplicates:	DUP (1 per 20 samples)
Reporting:	Maximum Significant Figures: 2
	W Value: 0.10 T Value: 0.50

CAEAL Accredited, QM Blind Audit participant

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 200.0 mg/L as BOD

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	125	200	203.560	3.560	9.2199
QCB:	125	100	102.420	2.420	4.5548
QCA+QCB:	125	300	305.984	5.984	12.8062
QCA−QCB:	125	100	101.136	1.136	6.8924

For 1993 Control Charts:

Sw (A−B) =   6.8697

Duplicates:

| Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
171	0 − 40	11.466	1.3741
64	40 − 100	70.453	4.1655
41	100 − 200	143.683	8.9912

Biochemical Oxygen Demand, QCA
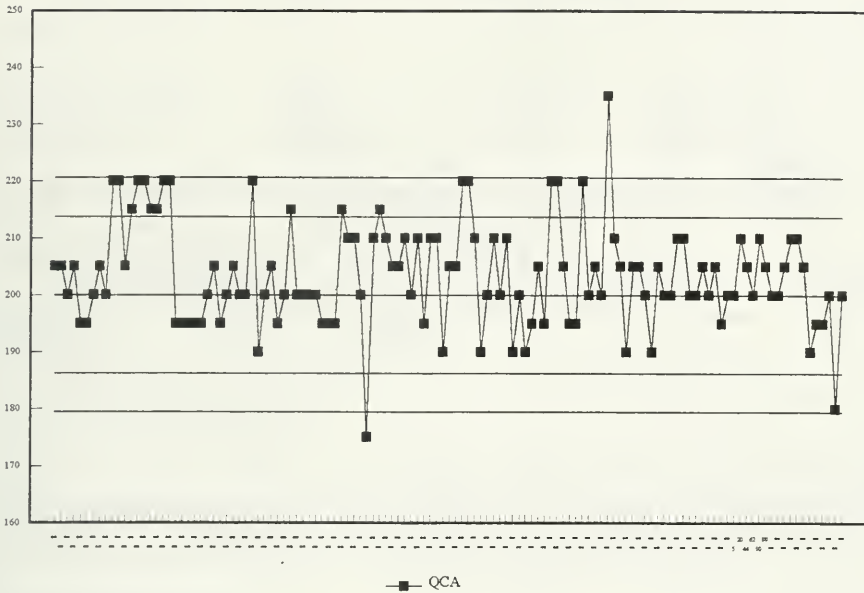
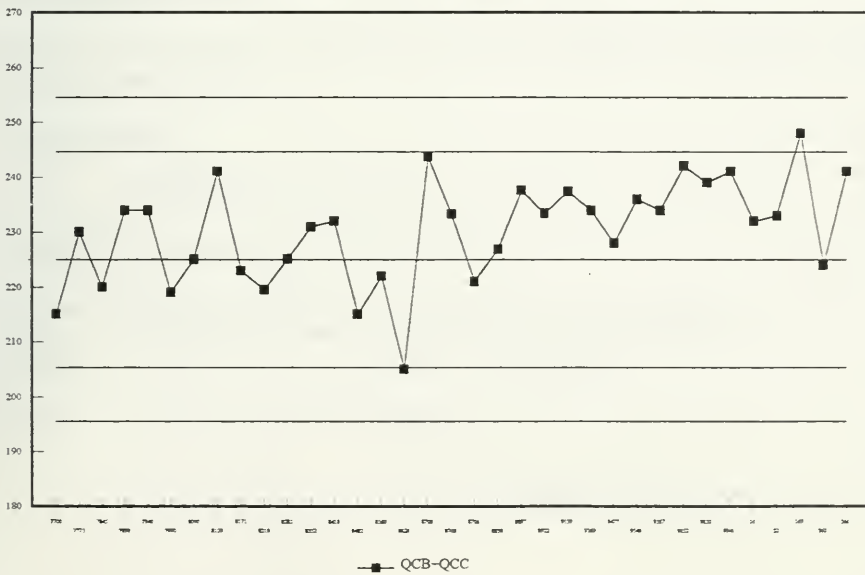# Biochemical Oxygen Demand, QCB
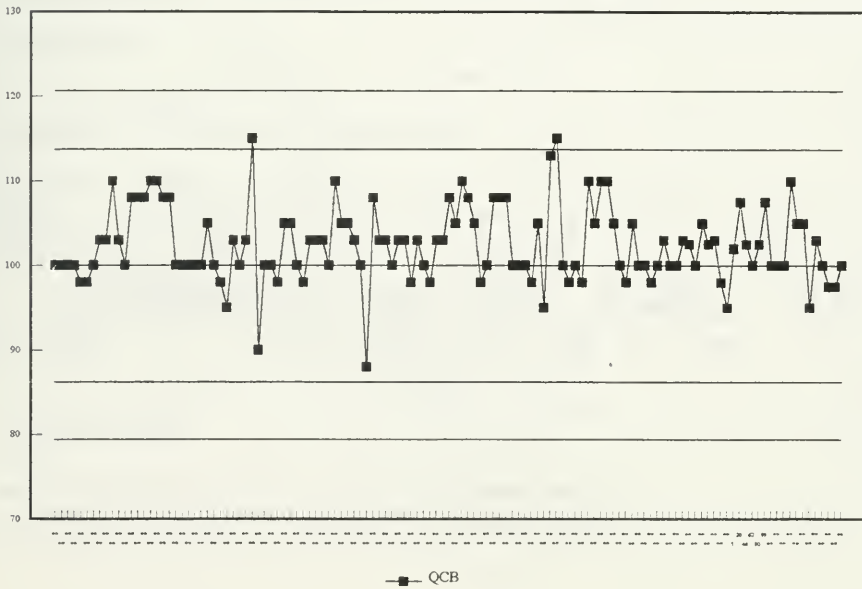
Biochemical Oxygen Demand, QC Sum
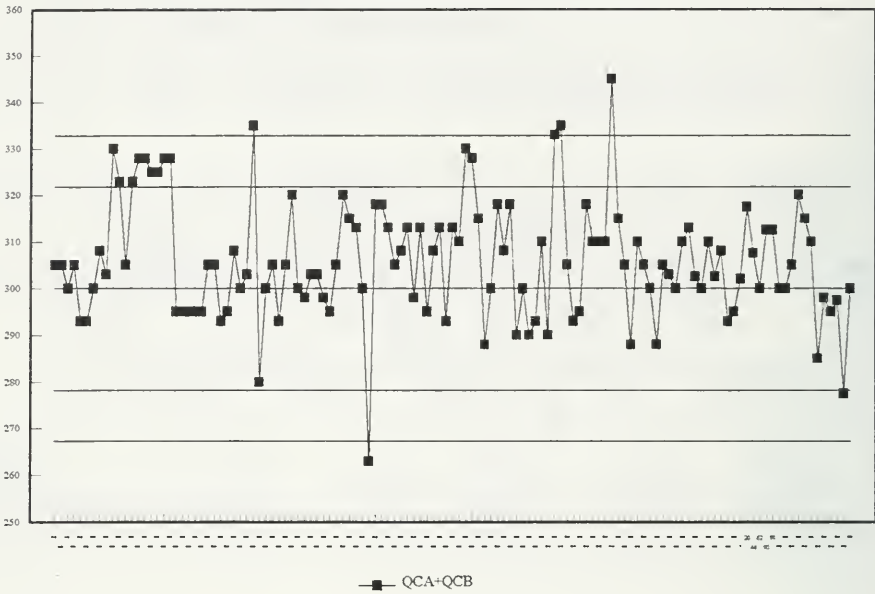
Biochemical Oxygen Demand, QC Difference

58
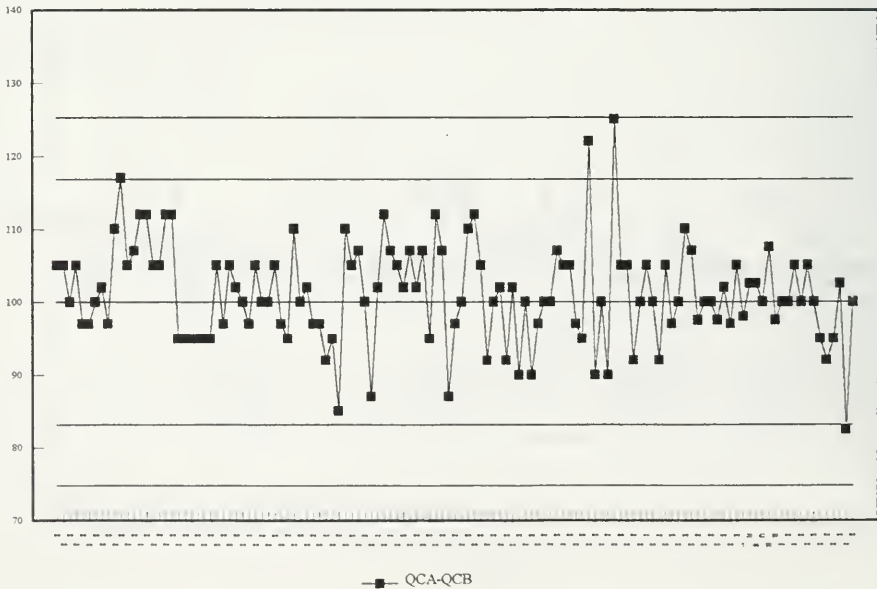
CHEMICAL OXYGEN DEMAND

IDENTIFICATION:

LIS Test Name Code:	COD	Introduced:	February, 1981
Work Station Code:	TBCOD	Unit:	mg/L as O_2
Method Code:	E6028A	Section:	Water Quality

SAMPLE TYPE/MATRIX: Sewage, leachates, industrial waste, surface and domestic waters.

SAMPLING:

Special Instructions:	Freeze sample if delays are unavoidable.
Container:	PET
Preservative:	Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Samples (5 or 10 mls) are mixed with acidified potassium dichromate which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a convection oven for 3 hours at 150°C. The digested standards and blanks are then read using a spectrophotometer.

INSTRUMENTATION:

Spectronic 20, set at a wavelength of 600 nm.

CALIBRATION: - Linear
   - 6 Standards, 20 - 900 mg/L COD

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA, QCB, QCC
Drift:	2 Undigested Blanks, ZERO %T check every 10 samples
Duplicates:	DUP (1 per 10 samples)

QM Blind Audit participant

Reporting:	Report to: 5-100 nearest 1, 100-1000 nearest 5
	W Value: 5 T Value: 25

MODIFICATIONS:

February, 1981 - Analysis using HAMES reflux method initiated
July, 1983 - COD analysis was updated to the present procedure.

CHEMICAL OXYGEN DEMAND

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 900.0 mg/L as O2

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	35	600	605.629	5.629	12.3743
QCB:	35	200	207.629	7.629	6.7086
QCC:	35	25	22.540	−2.460	5.5934
QCA+QCB:	35	800	813.257	13.257	17.2888
QCA−QCB:	35	400	398.000	−2.000	9.8668
QCB+QCC:	35	225	230.169	5.169	9.4722
QCB−QCC:	35	175	185.089	10.089	7.9285

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 10.9229$$
$$Sw\ (B-C) = \quad 7.5587$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
45	0 − 180	47.125	5.0180
7	180 − 450	374.000	7.7644
2	450 − 900	570.000	35.0892

Chemical Oxygen Demand, QCA
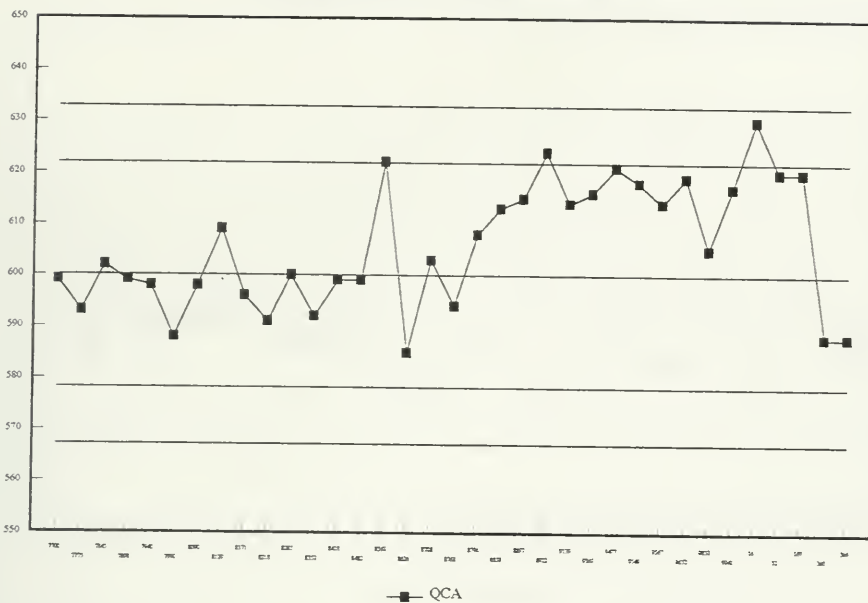
Chemical Oxygen Demand, QCB
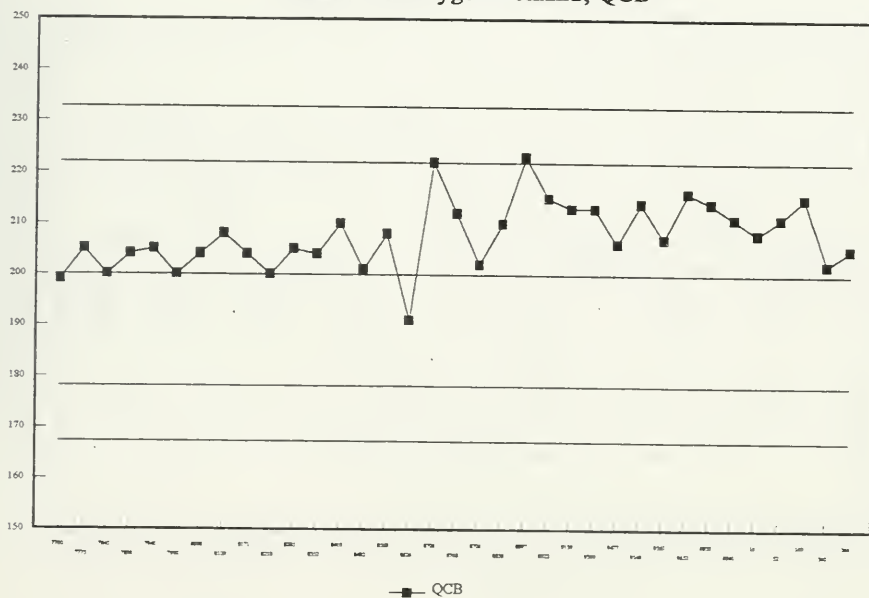
Chemical Oxygen Demand, QCC
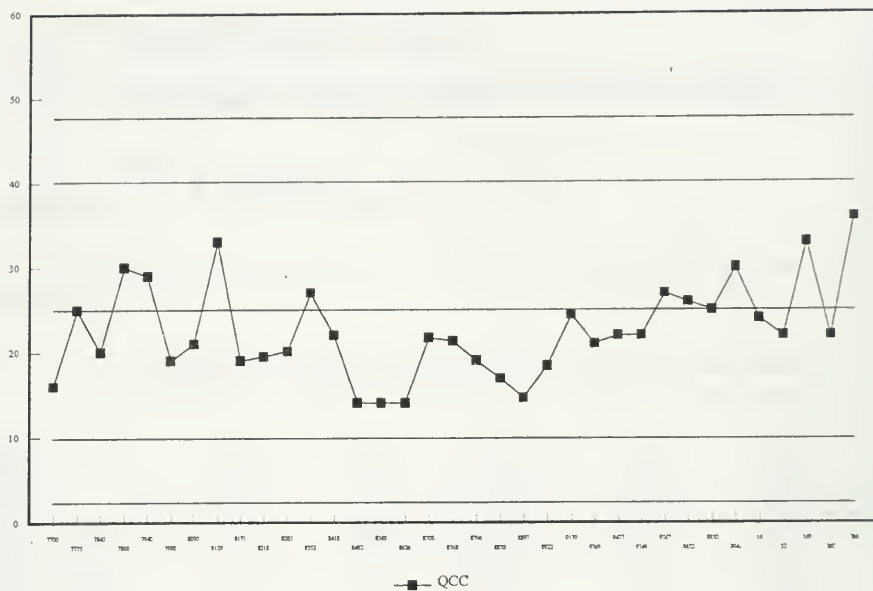
QCC

Chemical Oxygen Demand, QC Sum

QCA+QCB

62

Chemical Oxygen Demand, QC Difference

QCA-QCB
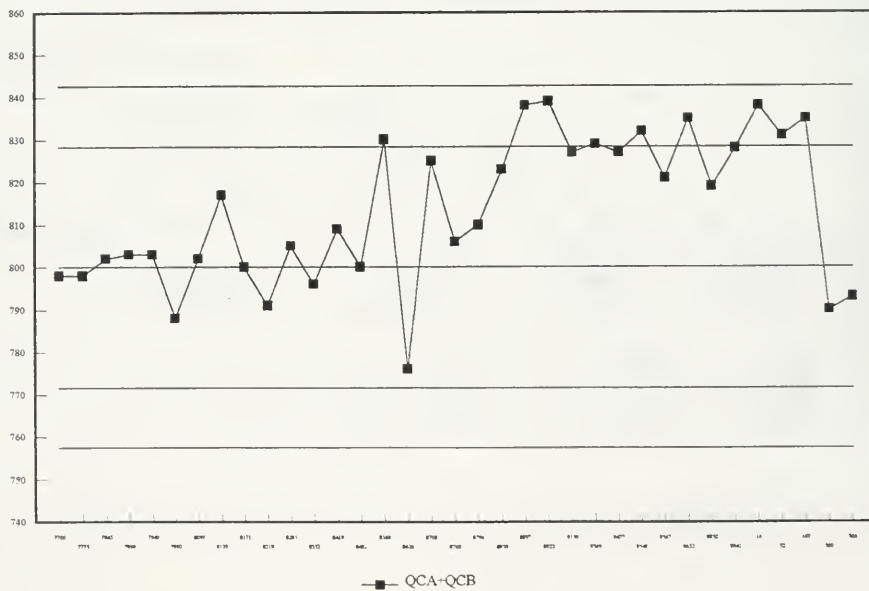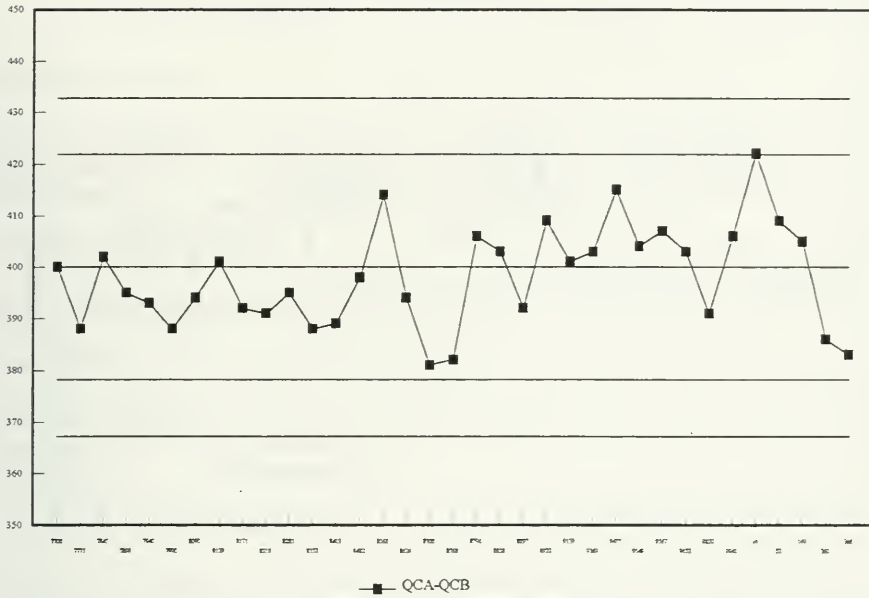
Chemical Oxygen Demand, QCC Sum

QCB-QCC

Chemical Oxygen Demand, QCC Difference

QCB-QCC
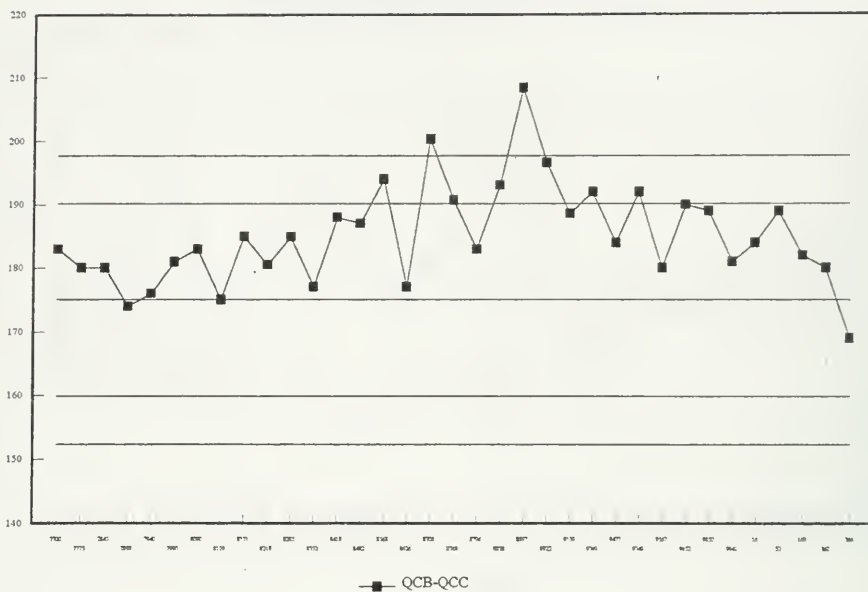
CHLORIDE

IDENTIFICATION:

LIS Test Name Code:	CLIDUR	Introduced:	January 1980
Work Station Code:	TBCLIDUR	Units:	mg/L as Cl
Method Code:	E6002A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, precipitation, sewage, industrial effluent and landfill leachate.

SAMPLING:

Special Instructions:
Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

Chloride ions combine with mercuric thiocyanate to form mercuric chloride and release thiocyanate ions to complex with the ferric ion producing a coloured solution - the absorbance of which is proportional to the concentration of chloride ion.

INSTRUMENTATION:

Technicon Auto-Analyzer II continuous flow system with colourimetric measurement through a 50mm light path at 460 nm.

CALIBRATION:
- Linear
- 10 Standards,   0-10 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB
Drift: BLK (every 10 samples), SENS.CHK, (2 every 20 samples)
Duplicates: DUP (1 for every 15 samples, run at beginning)

Reporting: Maximum Significant Figures: 2
               W Value: .05   T Value: 2.5

               CAEAL Accredited, LRTAP and QM Blind Audit participants

CHLORIDE

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 10.0 mg/L as Cl

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	71	8	7.994	−0.006	0.0402
QCB:	71	2	2.033	0.033	0.0337
QCA+QCB:	71	10	10.027	0.027	0.0581
QCA−QCB:	71	6	5.961	−0.039	0.0461

For 1993 Control Charts:

$$Sw\,(A-B) = \quad 0.0407$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
113	0 − 2	0.9198	0.0412
79	2 − 5	3.3550	0.0778
51	5 − 10	6.7004	0.0856

Chloride, QCA

QCA

Chloride, QCB

QCB

67
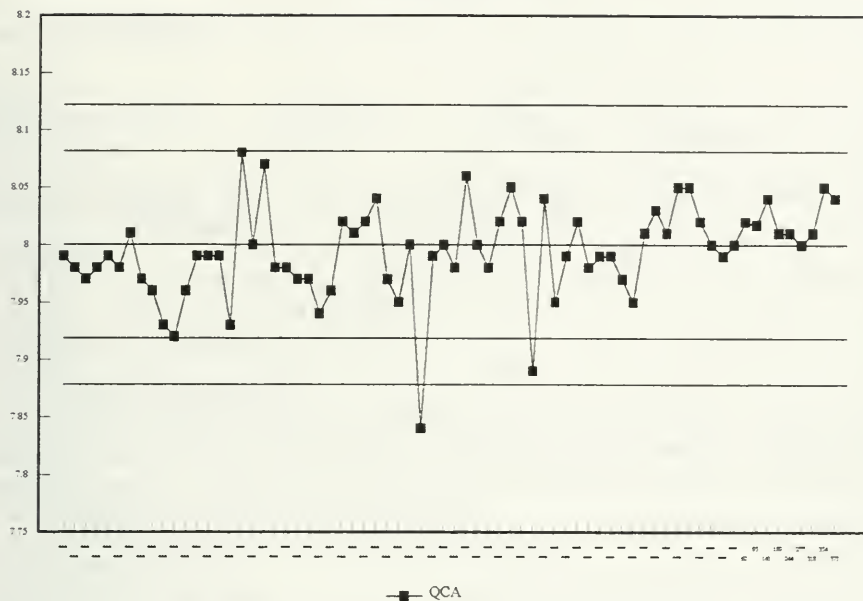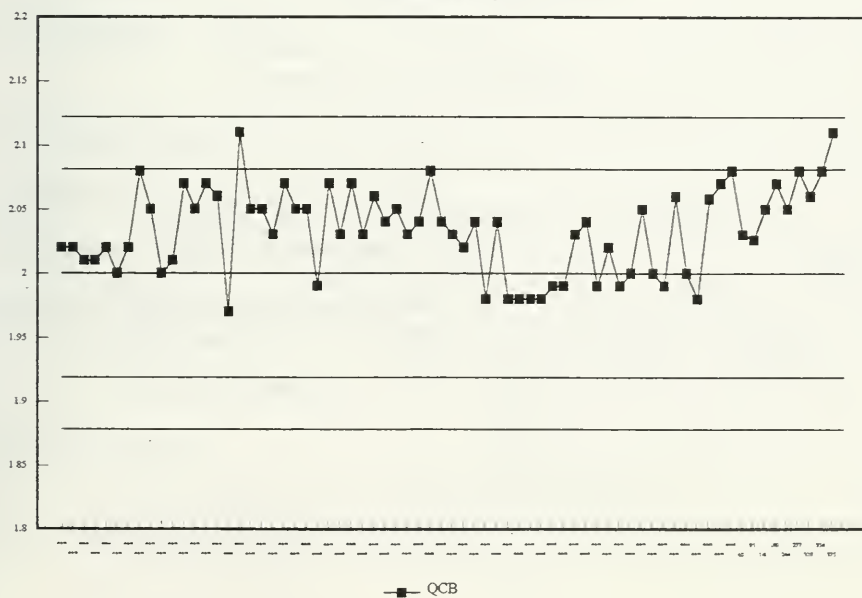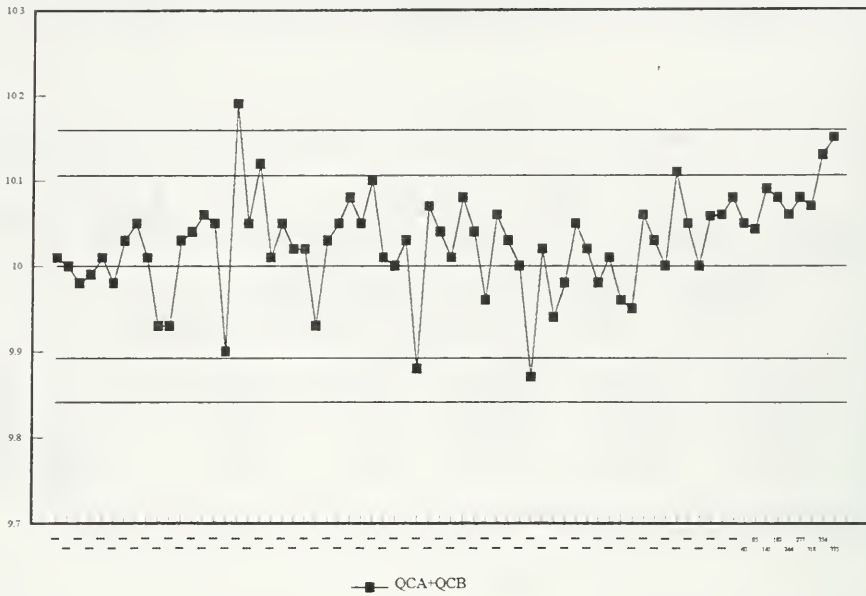
Chloride, QC Sum

Chloride, QC Difference

68
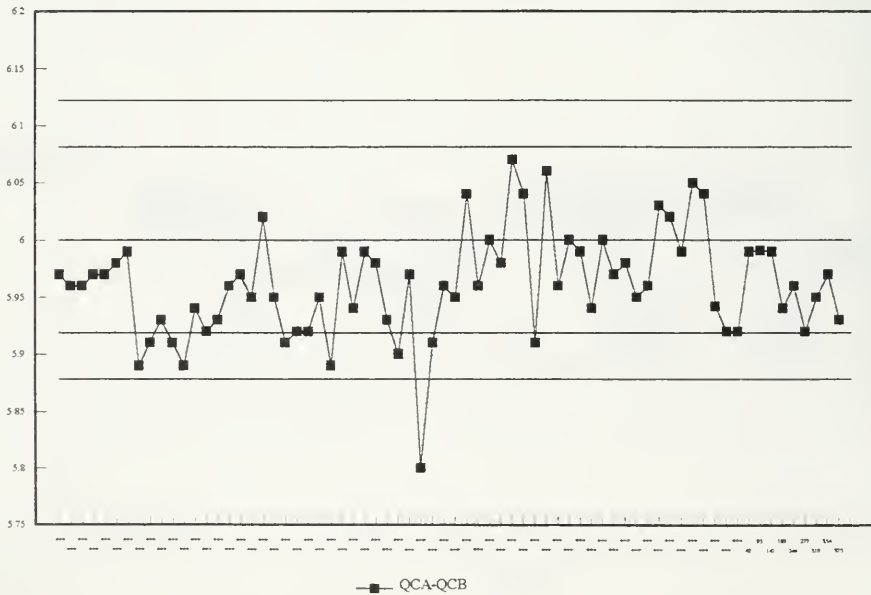
COLOUR - TRUE

IDENTIFICATION:

LIS Test Name Code:	COLTR	Introduced:	1978
Work Station Code:	TBCOLTR	Units:	TCU
Method Code:	E6004A	Section:	Water Quality

SAMPLE TYPE/MATRIX: Surface water, domestic water, leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerated at 4°C.

ANALYTICAL PROCEDURE:
True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

INSTRUMENTATION:
Automated continuous flow system, Technicon AAII. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450 nm.) Turbidity measurement is through a 5.0 cm. light path using a different broadband filter (660-740 nm).

CALIBRATION: - Linear
 - 4 Standards, 0 - 10.0 TCU range
 - 10 Standards, 2.0 - 80.0 TCU range

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: BLK every 10 samples, 10 and 60 STD every 20 samples
Duplicates: DUP (1 per 20 samples, run at beginning)

Reporting: Maximum Significant Figures: 2
 W Value: 0.5   T Value: 2.5

 LRTAP Participant

MODIFICATIONS:

Aug. 1985 - Apparent colour method upgraded to present method.
Feb. 1990 - Quality Control Standards changed from 2 samples to 3.

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 80.0 TCU

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	50	70	70.495	0.495	0.6010
QCB:	50	25	25.615	0.615	0.2932
QCC:	50	7	6.752	−0.248	0.2011
QCA+QCB:	50	95	96.110	1.110	0.8366
QCA−QCB:	50	45	44.881	−0.119	0.4409
QCB+QCC:	50	32	32.366	0.366	0.3916
QCB−QCC:	50	18	18.863	0.863	0.3153

For 1993 Control Charts:

$$Sw\,(A-B) = \quad 0.6019$$
$$Sw\,(B-C) = \quad 0.2413$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
107	0 − 16	6.612	0.4189
52	16 − 40	25.765	0.7019
28	40 − 80	53.053	0.9385

COLTR   Colour True
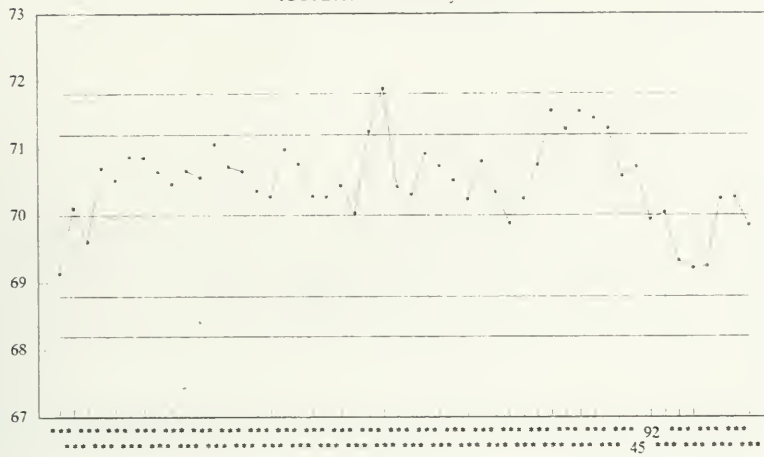
TBCOLTR   Thunder Bay Colour

_____ QCA

Jan. 01, 1993 – Dec. 31, 1993
Runs: 7720 – 376

COLTR   Colour True

TBCOLTR   Thunder Bay Colour

_____ QCB

Jan. 01, 1993 – Dec. 31, 1993
Runs: 7720 – 376

COLTR   Colour True
TBCOLTR   Thunder Bay Colour

COLTR   Colour True
TBCOLTR   Thunder Bay Colour

COLTR   Colour True
TBCOLTR   Thunder Bay Colour

QCA-QCB

Jan. 01, 1993 − Dec. 31, 1993
Runs: 7720 − 376

COLTR   Colour True
TBCOLTR   Thunder Bay Colour

QCB+QCC

Jan. 01, 1993 − Dec. 31, 1993
Runs: 7720 − 376

COLTR   Colour True
TBCOLTR   Thunder Bay Colour

QCB–QCC

Jan. 01, 1993 – Dec. 31, 1993
Runs: 7720 – 376

74
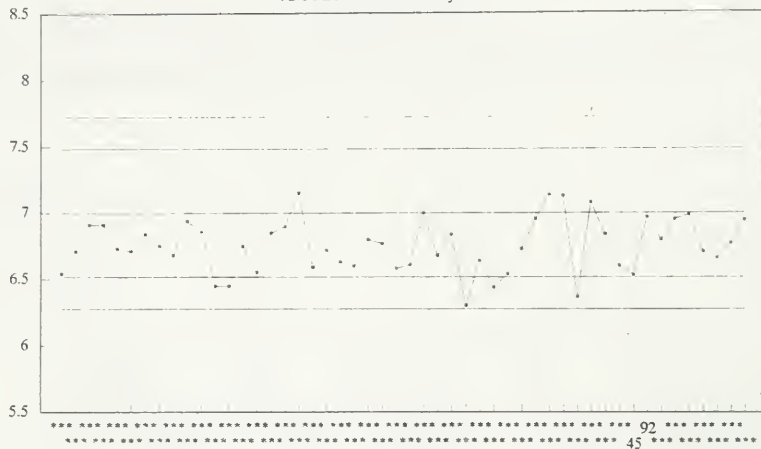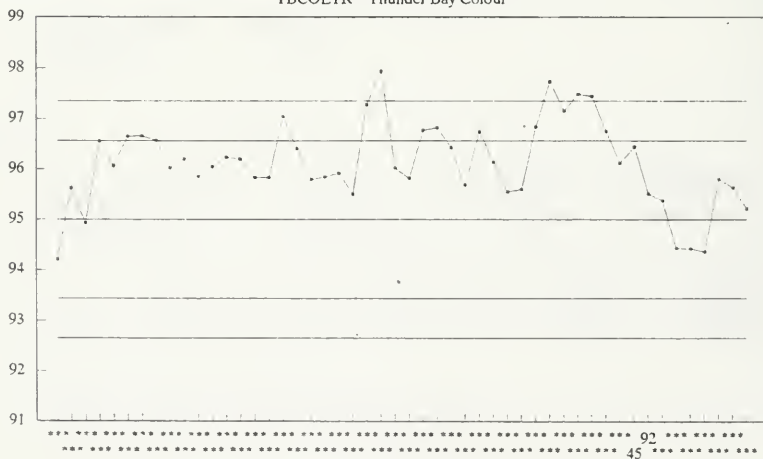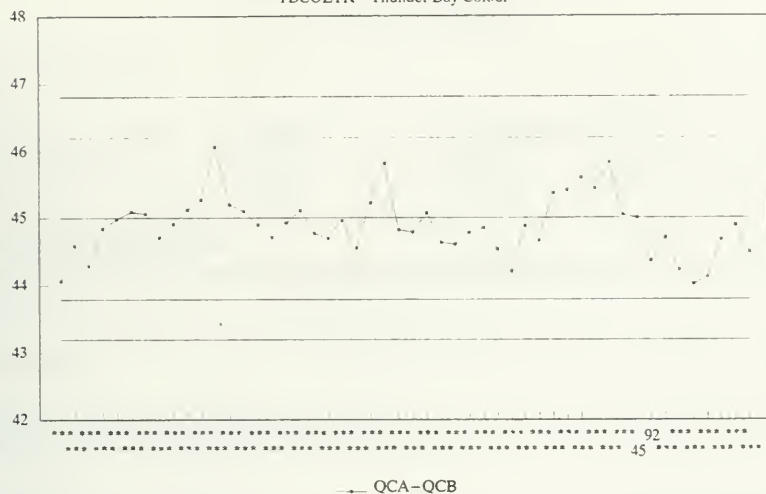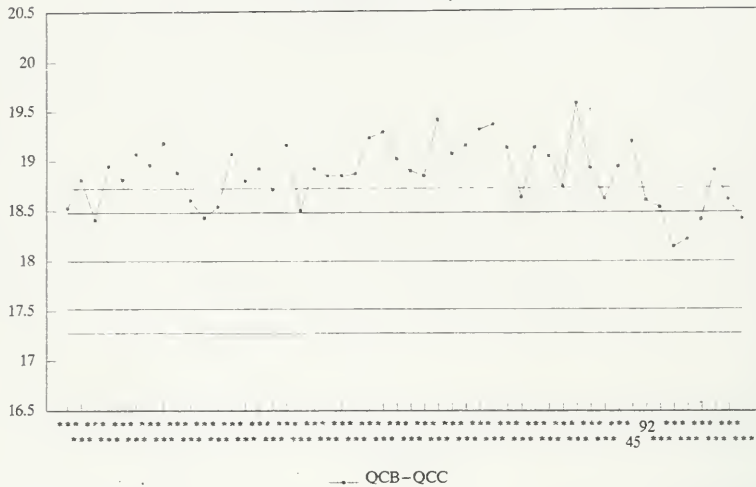
CONDUCTIVITY

IDENTIFICATION:

LIS Test Name Code:	COND25	Introduced:	1978
Work Station Code:	TBCAP	Units:	µS/cm
Method Code:	E6003A	Section:	Water Quality

SAMPLE TYPE/MATRIX:
Surface water, drinking water, ground water, sewage samples, landfill leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:
A conductivity cell is introduced into a sample at room temperature with continuous stirring. The conductivity is automatically printed out.

NOTE: Total Fixed Endpoint Alkalinity and pH are determined simultaneously.

INSTRUMENTATION:
Auto-Titration System, Radiometer, consisting of an ABU80 Autoburette, SAC80 Multisampler, TTT85 Titrator, CDM83 Conductivity Meter and a PRS12 Alpha Printer.

CALIBRATION:
1 Standard for Calibration set, 20 Standards for Calibration check.
Calibrated as required.

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: QC'S at end of run
Duplicates: DUP (1 every 10 samples)

        CAEAL Accredited, LRTAP participant

Reporting: Maximum Significant Figures: Whole Numbers
        W Value: 1   T Value: 5

MODIFICATIONS:

1986-Instrumentation changed from Radiometer CDM3
1990-LTB value is now subtracted from QC value.

CONDUCTIVITY

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 2000.0 uS/cm

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	188	718	718.451	0.451	3.8711
QCB:	188	147	149.767	2.767	1.3637
QCC:	188	74	75.432	1.432	0.5624
QCA+QCB:	188	865	868.218	3.218	4.7314
QCA−QCB:	188	571	568.684	−2.316	3.3622
QCB+QCC:	188	221	225.199	4.199	1.6779
QCB−QCC:	188	73	74.335	1.335	1.2398

For 1993 Control Charts:

$$Sw (A-B) = \quad 3.2866$$
$$Sw (B-C) = \quad 1.0872$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
393	0 − 400	154.038	20.5870
113	400 − 1000	631.354	6.5212
25	1000 − 2000	1417.760	16.4323

Conductivity, QCA

QCA

Conductivity, QCB

QCB
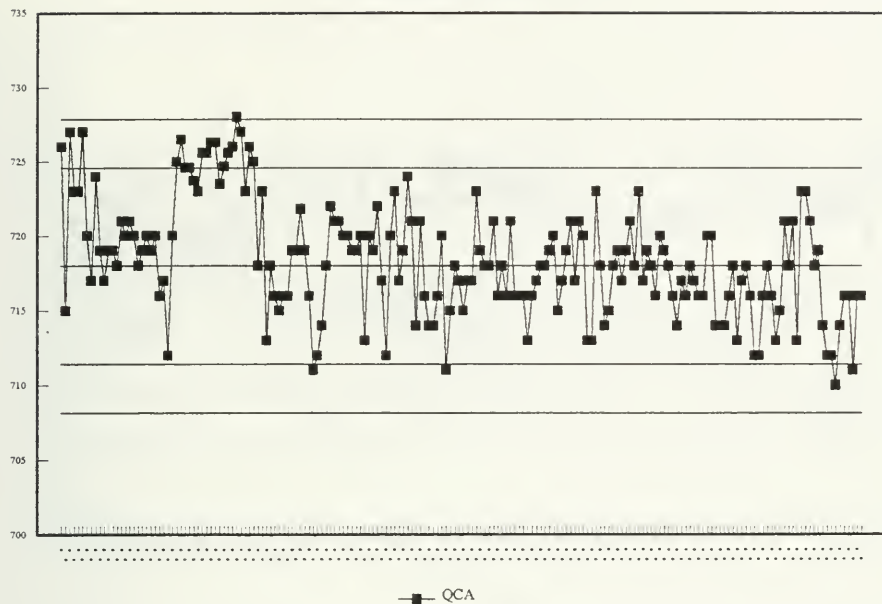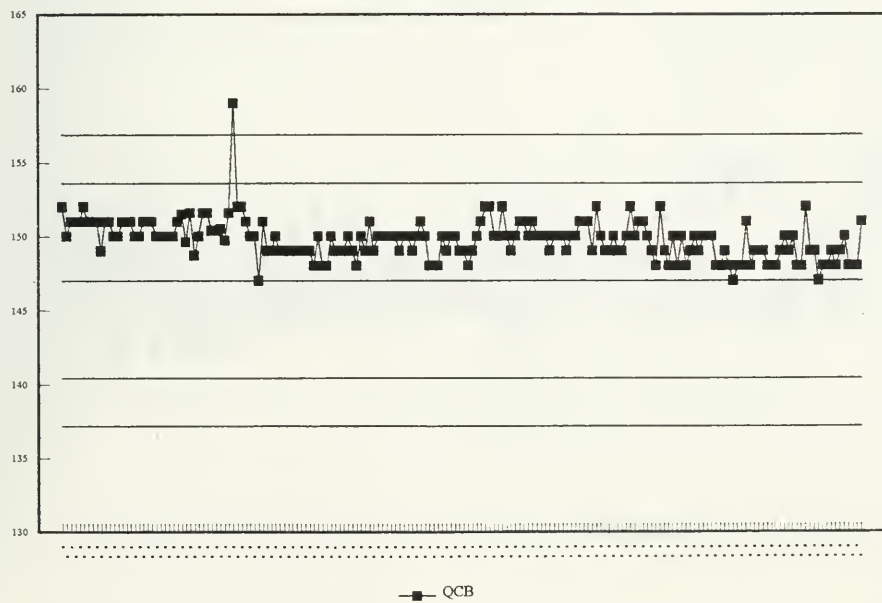
Conductivity, QCC

QCC
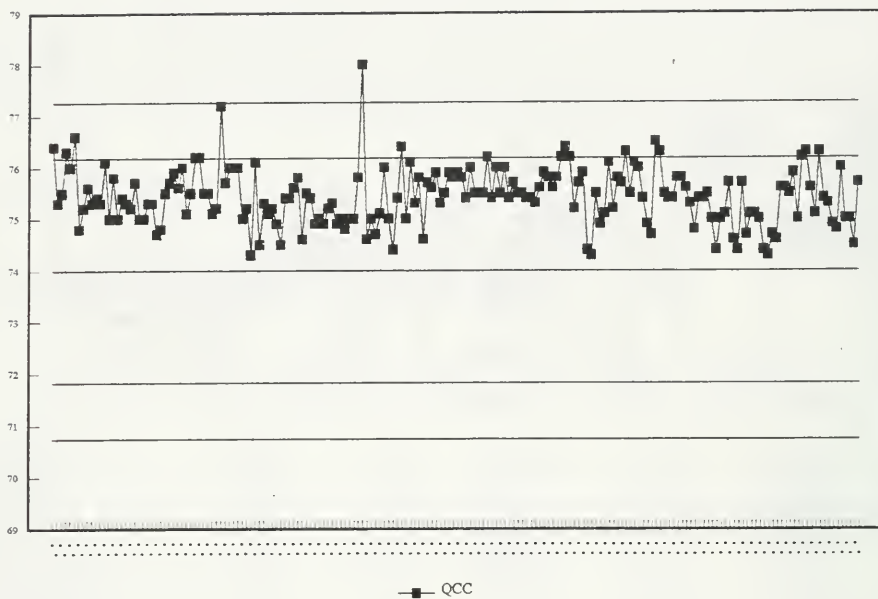
Conductivity, QC Sum

QCA+QCB

78
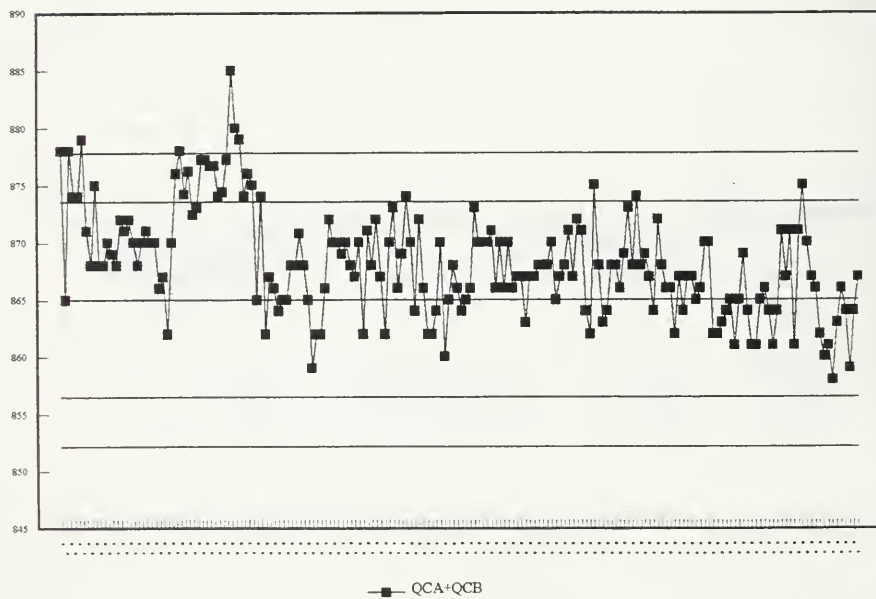
Conductivity, QC Difference

QCA-QCB
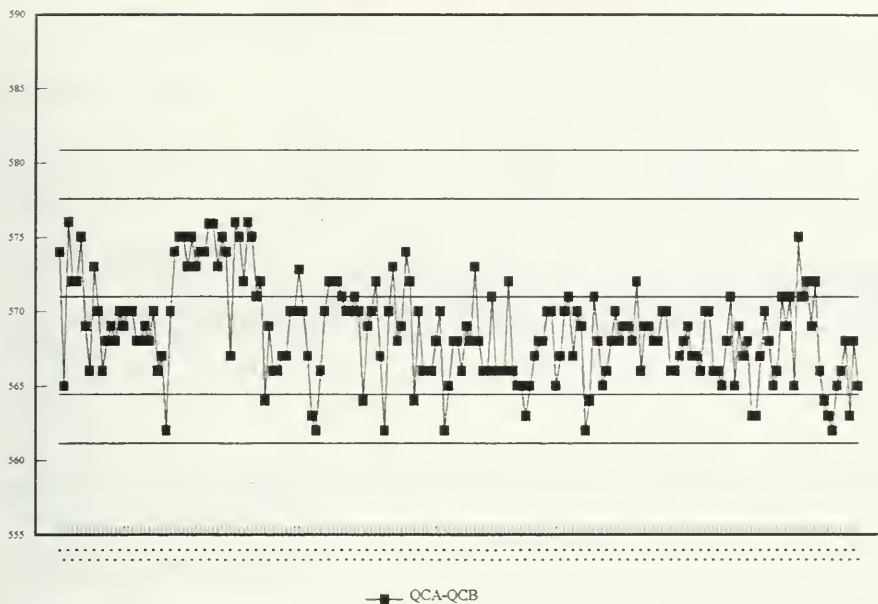
Conductivity, QCC Sum

QCB-QCC

79
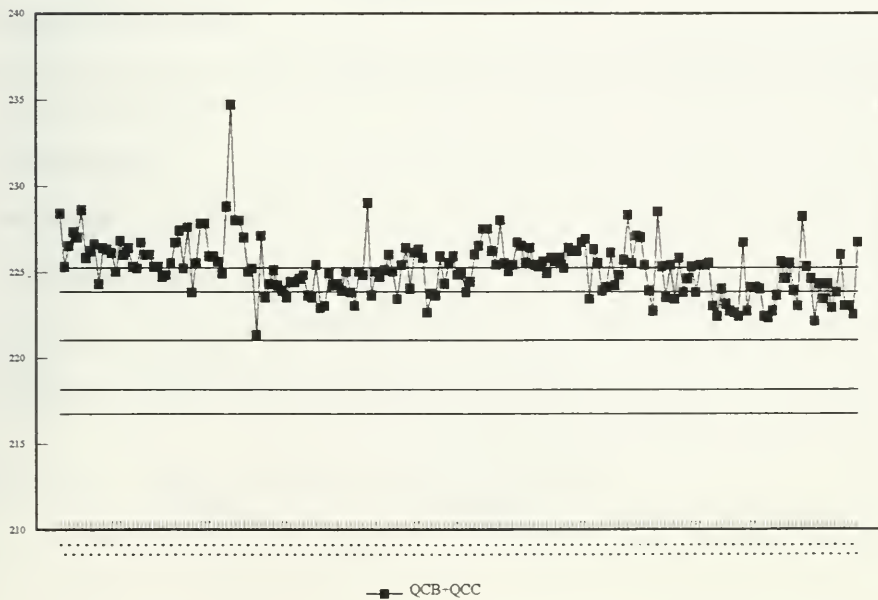
Conductivity, QCC Difference

QCB-QCC
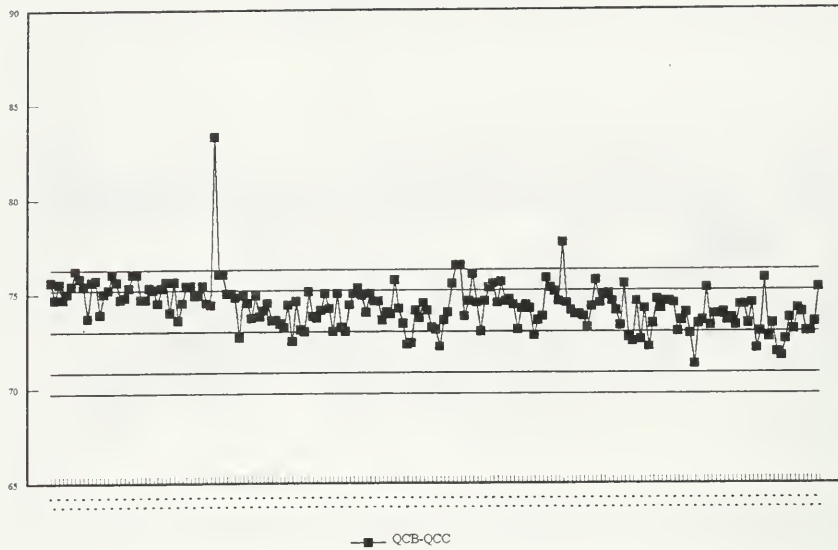
CONDUCTIVITY - CDM3

IDENTIFICATION:

LIS Test Name Code:	CDM3	Introduced:	May, 1977
Work Station Code:	TBCOND25	Units:	µS/cm
Method Code:	E6005A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, drinking water, ground water, sewage samples, landfill leachates and industrial effluents.

SAMPLING:

Special Instructions:
Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Sample is introduced into a jacketed conductivity cell and equilibrated to 25°C. The conductivity is read directly off the meter.

INSTRUMENTATION:

Radiometer Conductivity Meter Model CDM3 and a water jacketed cell temperature controlled by a water circulator.

CALIBRATION:

Predetermined cell constant.

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift:
Duplicates: DUP (1 every 10 samples)

Reporting: Maximum Significant Figures: Whole Numbers
              W Value: 1   T Value: 5

CONDUCTIVITY — CDM3

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 10,000.0 uS/cm

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation	
QCA:	19	718	715.158	−2.842	5.0029	
QCB:	19	147	147.000	0.000	2.6667	
QCC:	19	74	74.737	0.737	1.1945	
QCA+QCB:	19	865	862.158	−2.842	6.5512	
QCA−QCB:	19	571	568.158	−2.842	4.6220	**
QCB+QCC:	19	221	221.737	0.737	3.3804	
QCB−QCC:	19	73	72.263	−0.737	2.3768	**

For 1993 Control Charts:

Sw (A−B) =   1.8708
Sw (B−C) =   1.4814

**Note: This instrument was sent for repairs April 1993, therefore previous standard deviations will be used for 1994 graphs.

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
6	0 − 2000	865	5.4924
1	2000 − 5000	4025	0.0000
1	5000 − 10000	9430	21.2132

Conductivity-CDM3, QCA

x-axis labels: 7699 7699 0 7812 7812 8110 8152 8179 8213 8278 8401 8409 8437 8522 8776 8849 0 9875 0

Legend: QCA

Conductivity-CDM3, QCB

x-axis labels: 7699 7699 0 7812 7812 8110 8152 8179 8213 8278 8401 8409 8437 8522 8776 8849 0 9875 0
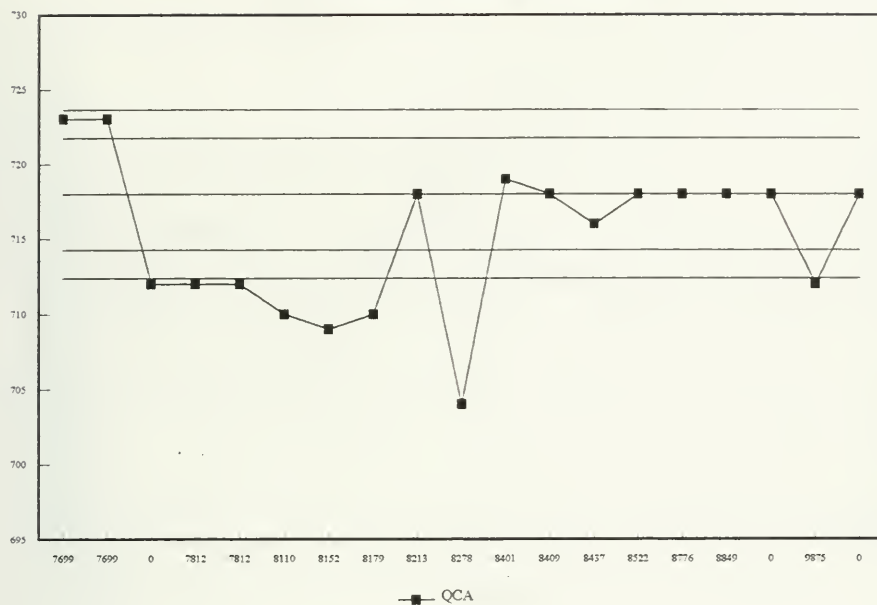
Legend: QCB
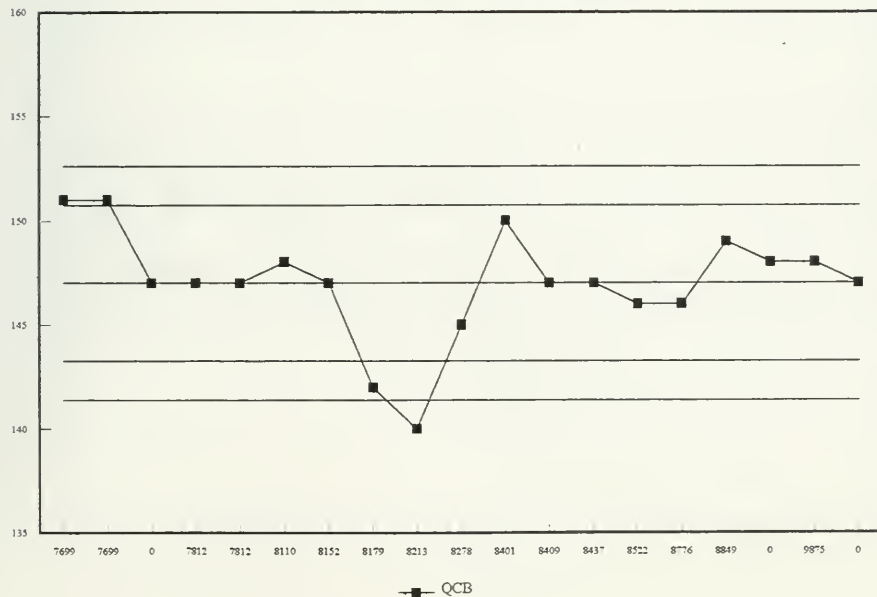
Conductivity-CDM3, QCC
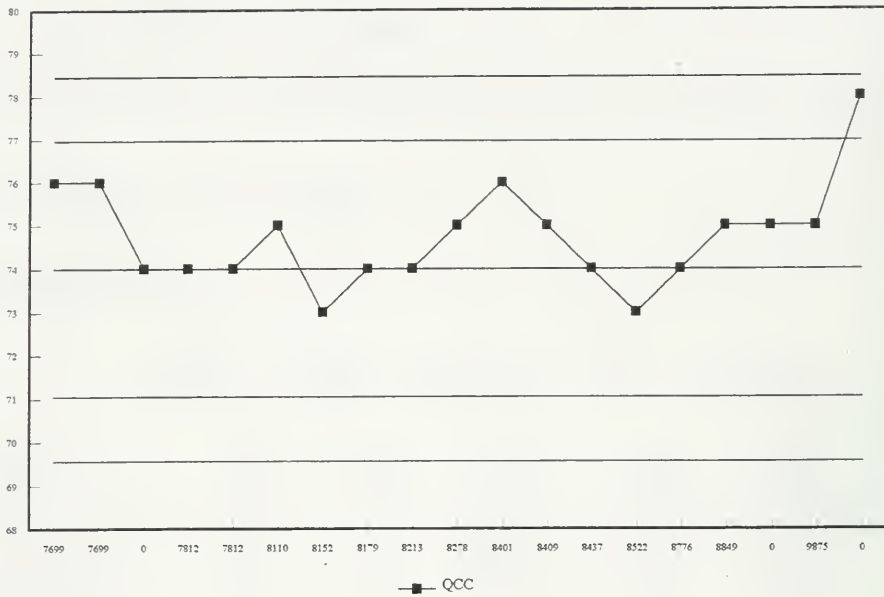
Conductivity-CDM3, QC Sum
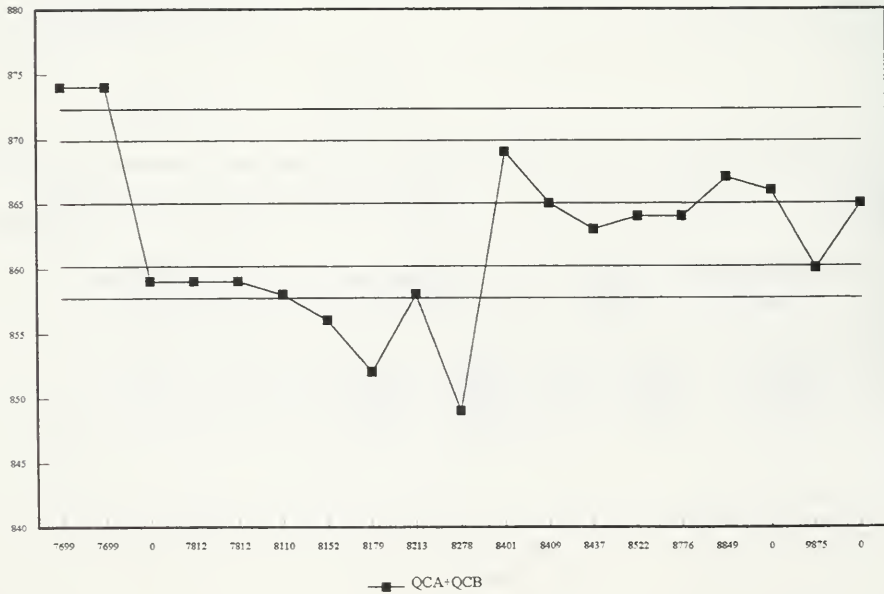
Conductivity-CDM3, QC Difference
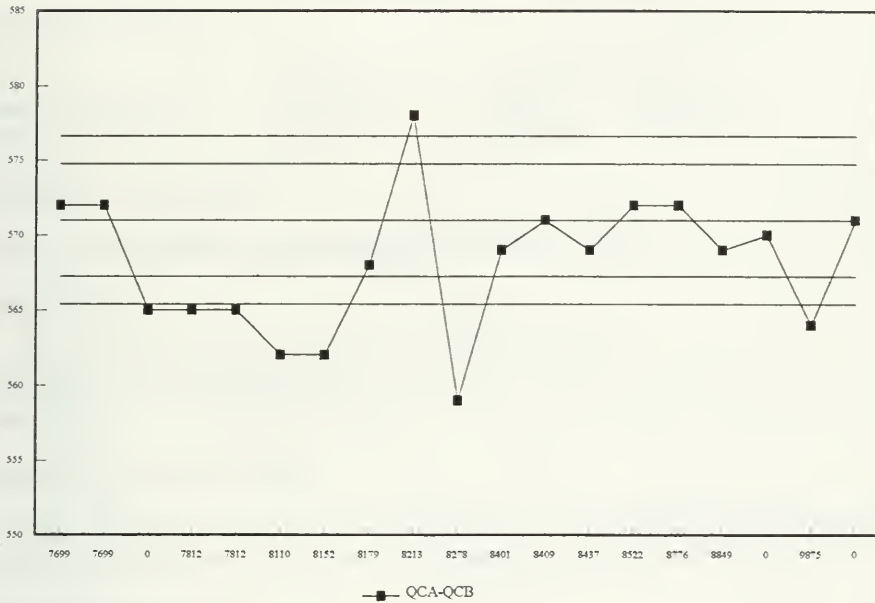
Conductivity-CDM3, QCC Sum
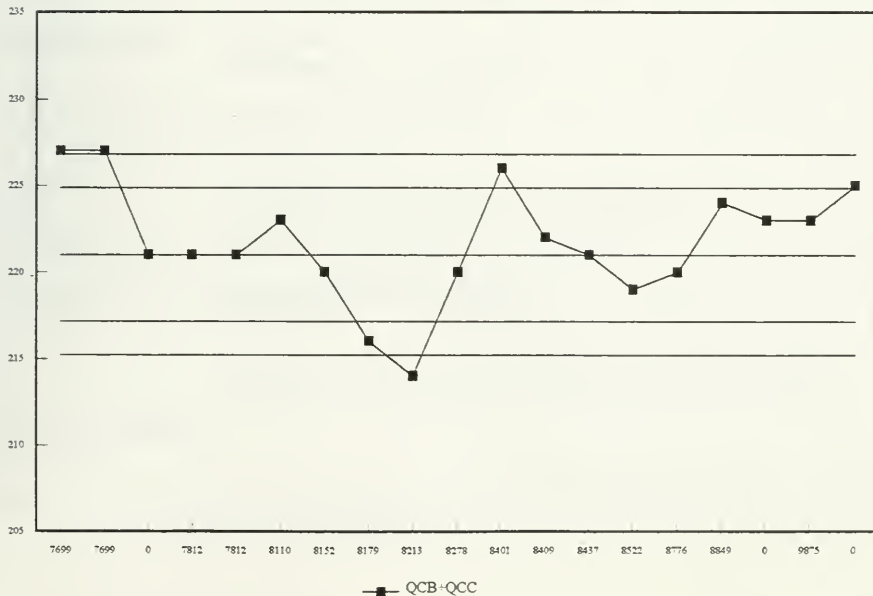
Conductivity-CDM3, QCC Difference
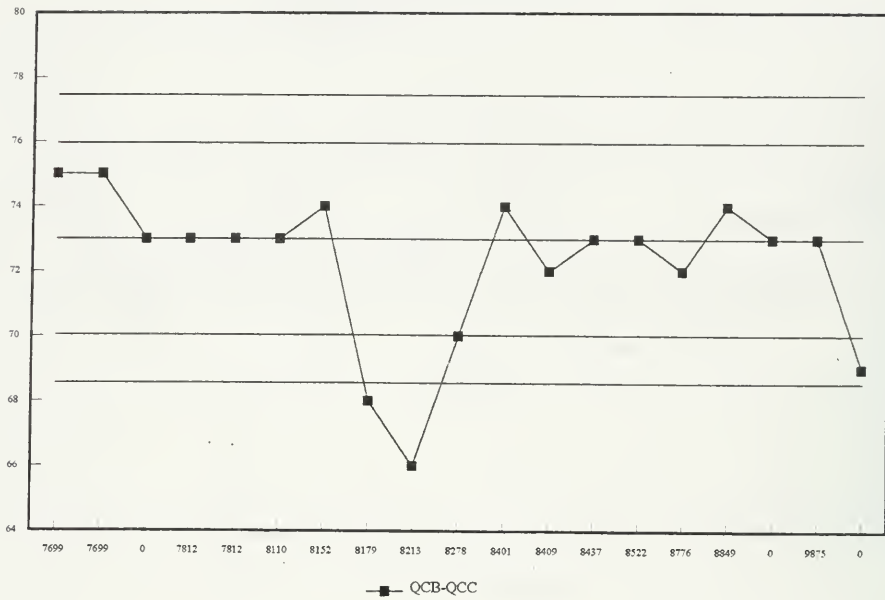
FLUORIDE

IDENTIFICATION:

LIS Test Name Code:	FFIDUR	Introduced:	1978
Work Station Code:	TBFFIDUR	Units:	mg/L as F
Method Code:	E6007A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, precipitation and landfill leachates.

SAMPLING:

Special Instructions:	No glass bottles if high level expected.
Container:	PET or glass
Preservative:	Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Fluoride is determined by an ion specific electrode. Any complexed fluoride in the sample is dissociated by the addition of a Total Ionic Strength Adjustment Buffer (TISAB).

INSTRUMENTATION:

Radiometer Ion85 Ion Analyzer, with SAC80 Automated Sample Changer.

CALIBRATION:
- Logarithmic
- 5 Standards, 0.1 - 2.0 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA, QCB
Drift:	0.2 and 2.0 mg/l standards every 20 samples
Duplicates:	1 for every 15 samples, run at beginning

Reporting:	Maximum Significant Figures: 2
	W Value: .01 T Value: .05

CAEAL Accredited, LRTAP and QM Blind Audit participants

MODIFICATIONS:

April 1988- The method was changed to the present automated system from an Ion Specific electrode and pH/Ion meter.

FLUORIDE

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 2.0 mg/L as F

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	59	1.4	1.402	0.002	0.0265
QCB:	59	0.4	0.398	−0.002	0.0247
QCA+QCB:	59	1.8	1.801	0.001	0.0360
QCA−QCB:	59	1.0	1.004	0.004	0.0364

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.0309$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
112	0.0 – 0.4	0.108	0.0210
13	0.4 – 1.0	0.789	0.0480
18	1.0 – 2.0	1.249	0.0420

Fluoride, QCA

■ QCA

Fluoride, QCB

■ QCB

89

Fluoride, QC Sum

QCA+QCB
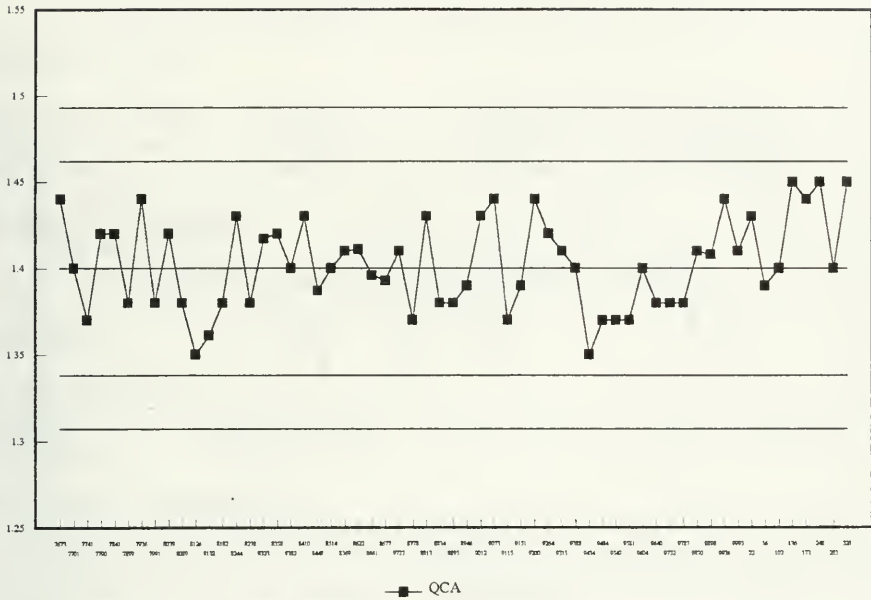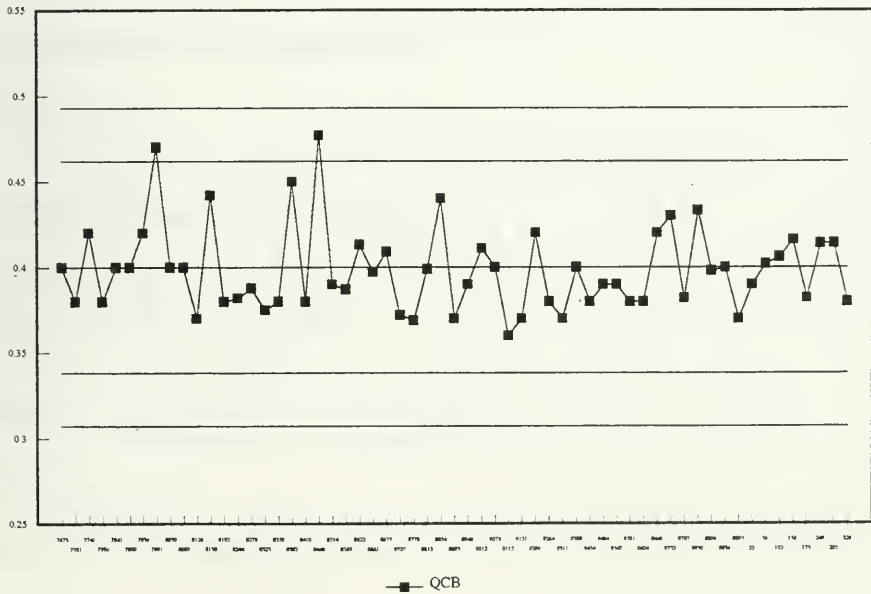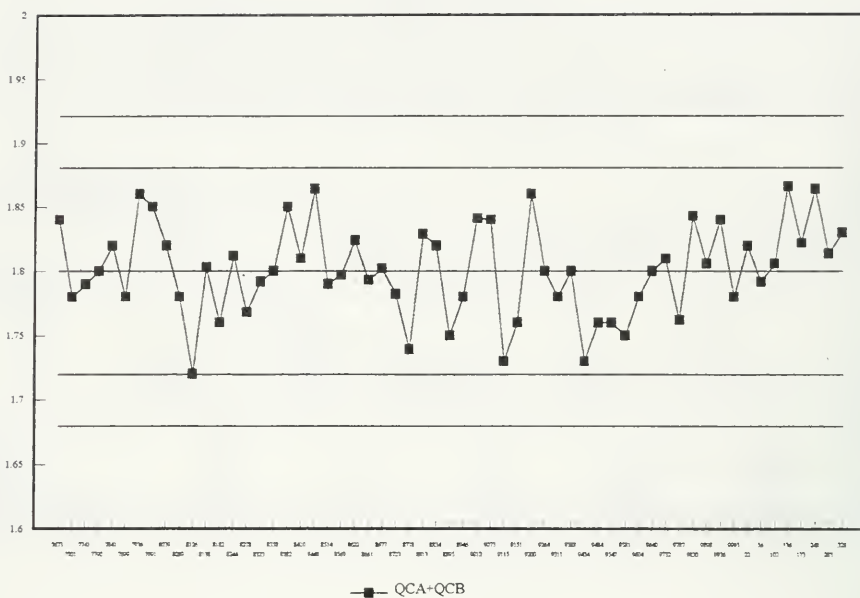
Fluoride, QC Difference

QCA-QCB
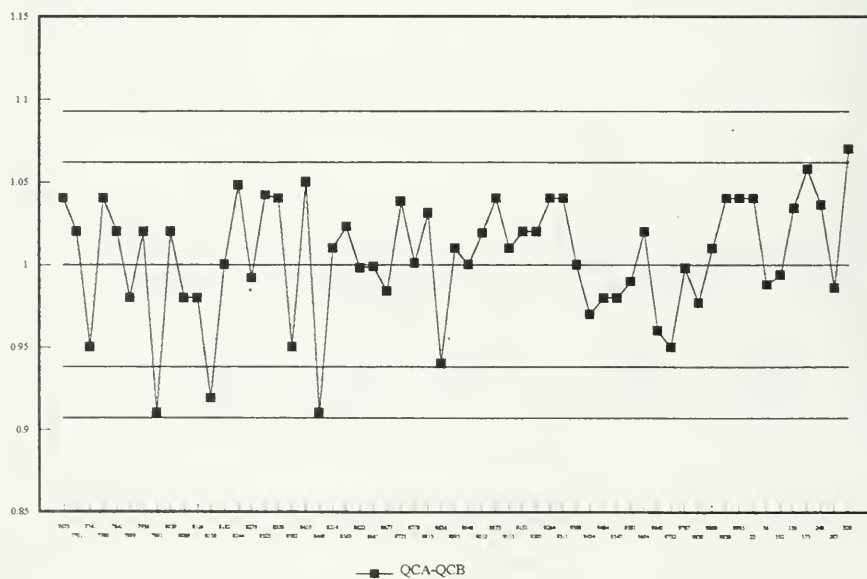
HARDNESS

IDENTIFICATION:

LIS Test Name Code:	HARDT	Introduced:	May, 1981
Work Station Code:	TBHARD	Units:	mg/L as $CaCO_3$
Method Code:	E6008A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, landfill leachates and industrial effluents.

SAMPLING:

Special Instructions:
Container: Glass or PET jar.
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

A manual titrimetric method is used where Erichrome Black T is added to an aliquot of sample and is buffered to pH 10. The resulting wine-red coloured solution is then titrated with EDTA, a chelating agent, to a blue end-point.

INSTRUMENTATION:

Brinkmann Auto-Burette with digital display.

CALIBRATION:

- EDTA standardized with Calcium Carbonate Standard Solution

CONTROLS AND QUALITY ASSURANCE:

Calibration: QCA, QCB
Drift:
Duplicates: DUP (1 per 20 samples)

Reporting: Maximum Significant Figures: 2
               W Value: .5   T Value: 2.5

MODIFICATIONS:

May, 1986 - A digital burette was incorporated.

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 200.0 mg/L as CaCO3

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	106	50	50.583	0.583	0.7596
QCB:	106	25	25.391	0.391	0.5023
QCA+QCB:	106	75	75.974	0.974	0.9897
QCA−QCB:	106	25	25.192	0.192	0.8244

For 1993 Control Charts:

$$Sw (A-B) = \quad 0.7525$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
73	0 – 40	22.111	0.6819
86	40 – 100	69.247	1.2081
49	100 – 200	148.020	1.9431

Hardness, QCA

QCA

Hardness, QCB

QCB

93

Hardness, QC Sum

QCA+QCB
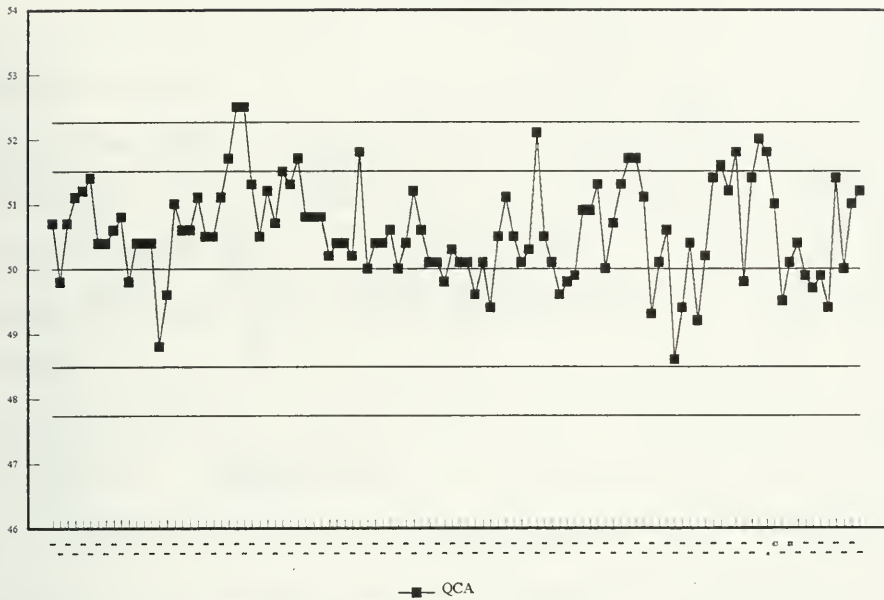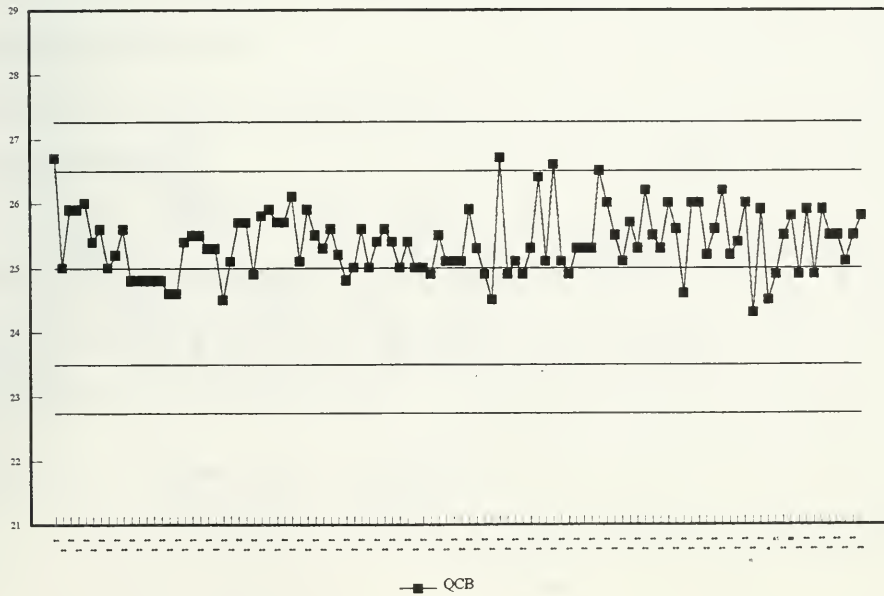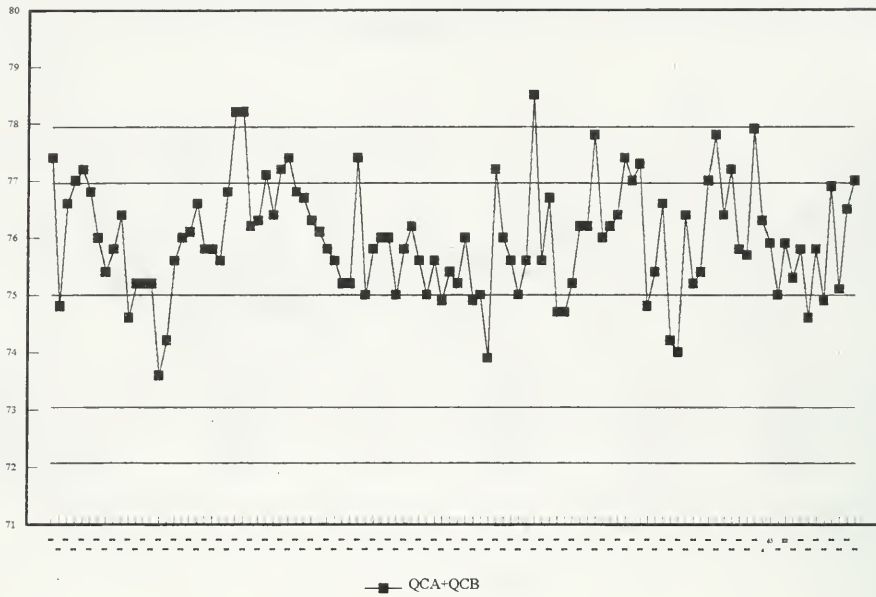
Hardness, QC Difference

QCA-QCB

94
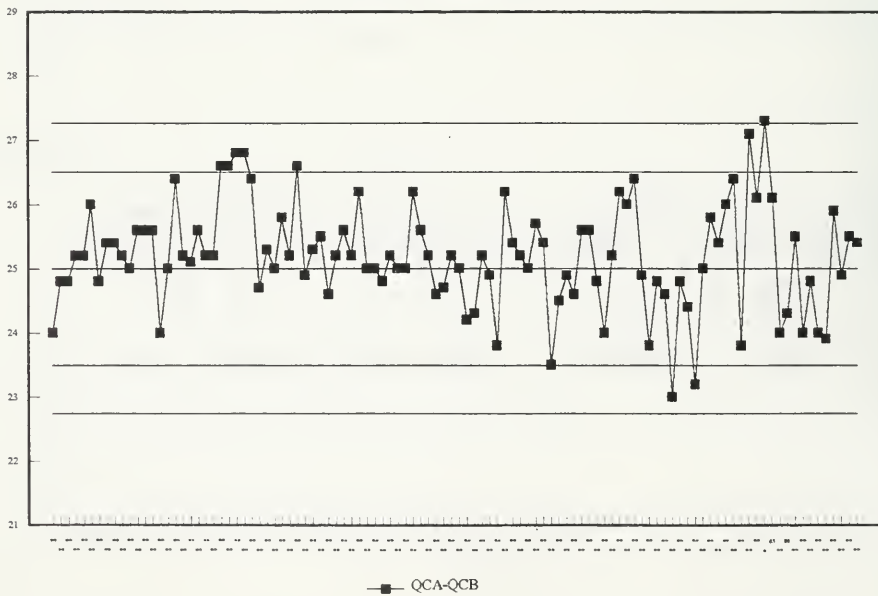
IRON - COLOURMETRIC

IDENTIFICATION:

LIS Test Name Code:	FEUTW	Introduced:	May, 1978
Work Station Code:	TBFEUTW	Units:	mg/L as Fe
Method Code:	E6006A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, ground water and industrial effluents.

SAMPLING:

Special Instructions:
Container: · Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

An aliquot of sample, usually 50 mls is mixed with an acidified hydroxylamine hydrochloride solution and concentrated. Ortho-phenanthroline and a buffer are added causing a colour development. The coloured solution formed obeys Beer's Law and its intensity is measured using a spectrophotometer.

INSTRUMENTATION:

Spectronic 20, set at a wavelength of 510 nm.

CALIBRATION:
           - Linear
           - 10 Standards, .20 - 2.00 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: DIG. BLANKS, QCA, QCB, QCC
Drift: 100 %T check DDW, ZERO %T check every 10 samples
Duplicates: DUP (1 per 10 samples)

Reporting: Maximum Significant Figures: 2
               W Value: 0.01   T Value: 0.05

IRON — COLOURMETRIC

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 2.0 mg/L as Fe

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	43	1	1.019	0.019	0.0200
QCB:	43	0.2	0.195	−0.005	0.0112
QCA+QCB:	43	1.2	1.213	0.013	0.0259
QCA−QCB:	43	0.8	0.823	0.023	0.0195

For 1993 Control Charts:

Sw (A−B) =   0.0193

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
75	0.0 − 0.4	0.091	0.0144
10	0.4 − 1.0	0.703	0.0172
2	1.0 − 2.0	1.420	0.0150

Iron-Dig. Colorimetric, QCA

Iron-Dig. Colorimetric, QCB
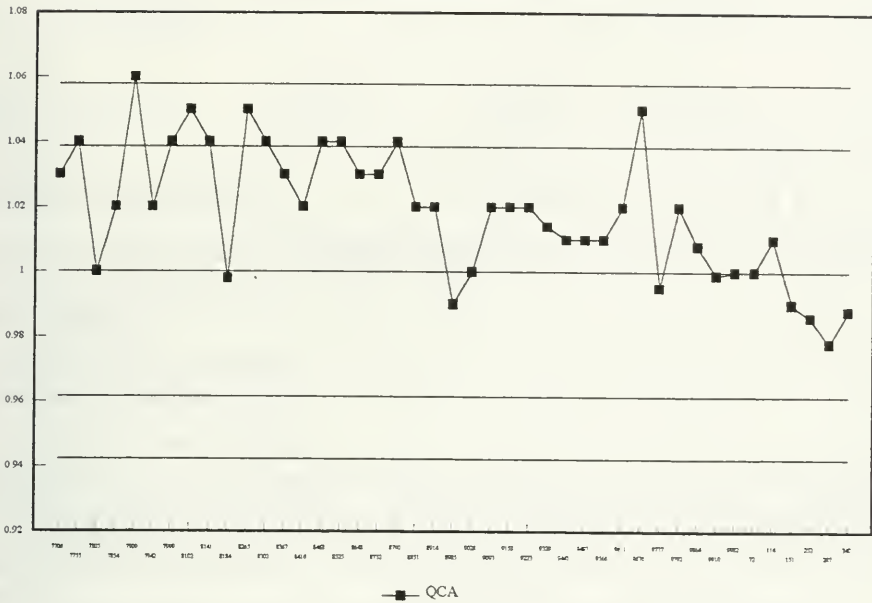
Iron-Dig. Colorimetric, QC Sum

■ QCA+QCB
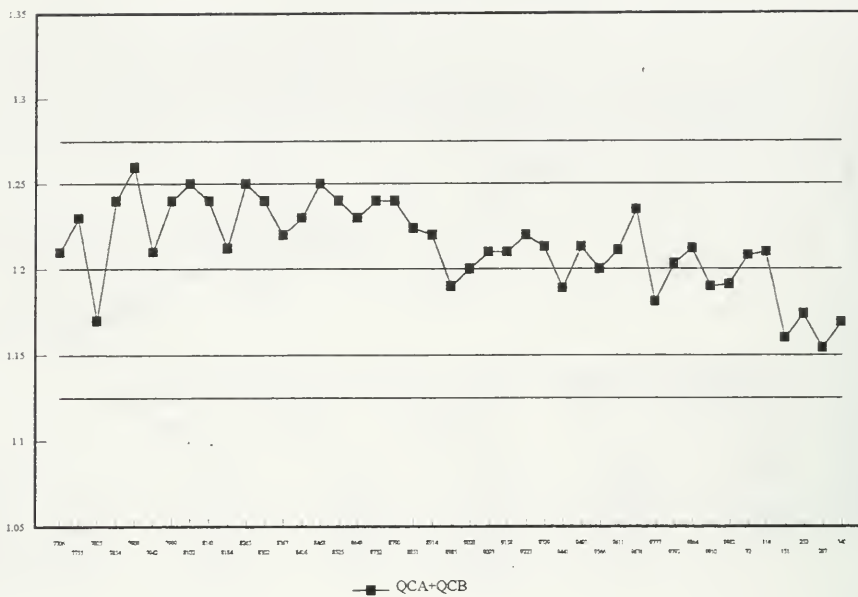
Iron-Dig. Colorimetric, QC Difference

■ QCA-QCB
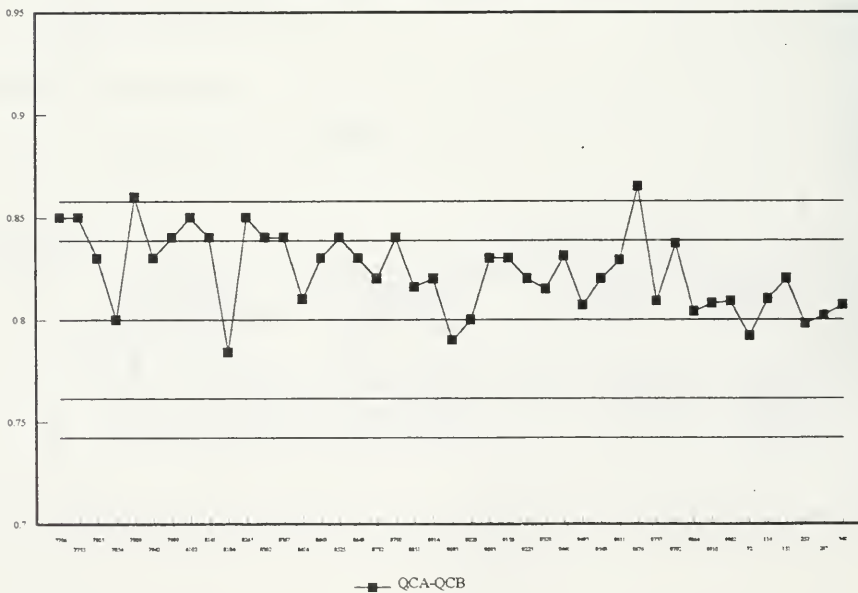
NITRATE

IDENTIFICATION:

LIS Test Name Code:	NNO3FR	Introduced:	1978
Work Station Code:	TBNDNP	Units:	mg/L as N
Method Code:	E6024A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:
Nitrate is reduced to nitrite by heating an aliquot of sample with hydrazine in alkaline media; this reaction is catalyzed by the addition of cupric ion. Subsequently, an azo dye is formed in acid media by diazotizing sulphanilamide with nitrite and coupling the product with N(1-naphthyl) ethylenediamine dihydrochloride. The nitrite result is subtracted from this nitrate plus nitrite reaction.
N.B. Ammonia, nitrite and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:
Automated continuous flow system, Technicon AAII with a 37°C heating bath. Colourimetric measurement is through a 5.0 cm. light path at 520 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION: - Linear
- 7 Standards   0 - 0.50 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA, QCB, QCC
Drift:	BLK every 10 samples, CHK (100%) every 20 samples
Duplicates:	DUP (3 per run, run at beginning)

Reporting:	Maximum Significant Figures: 3
	W Value: 0.01 T Value: 0.05

CAEAL Accredited, LRTAP and QM Blind Audit participant

MODIFICATIONS:
1988 - All channels went to microcomputer control with DCI software.

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 0.50 mg/L as N

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	84	0.320	0.321	0.001	0.0043
QCB:	84	0.080	0.080	0.000	0.0019
QCC:	84	0.032	0.032	0.000	0.0012
QCA+QCB:	84	0.400	0.401	0.001	0.0055
QCA−QCB:	84	0.240	0.241	0.001	0.0039
QCB+QCC:	84	0.112	0.111	−0.001	0.0027
QCB−QCC:	84	0.048	0.048	0.000	0.0015

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.0040$$
$$Sw\ (B-C) = \quad 0.0022$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
120	0.00 − 0.10	0.028	0.0040
40	0.10 − 0.25	0.165	0.0034
43	0.25 − 0.50	0.336	0.0066

Nitrate, QCA

QCA

Nitrate, QCB

QCB

Nitrate, QCC

QCC

Nitrate, QC Sum

QCA+QCB

102
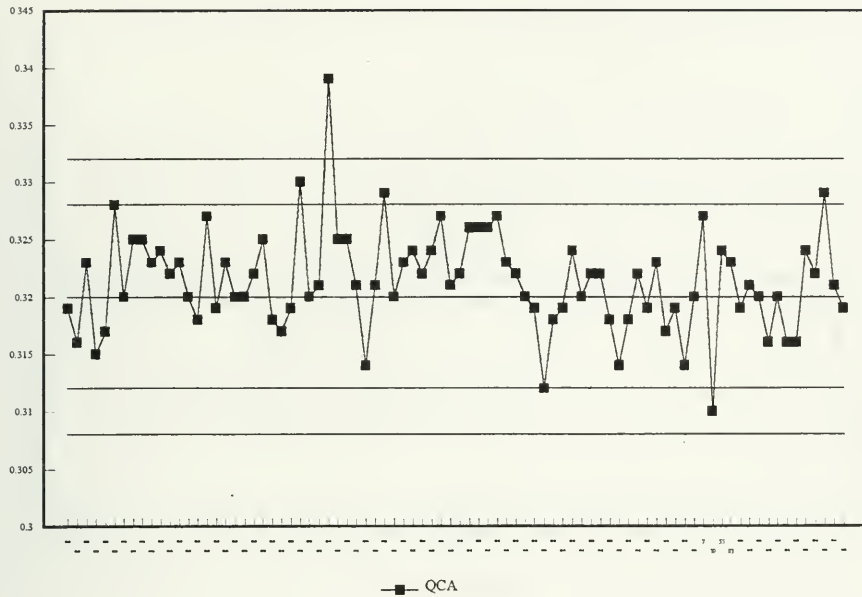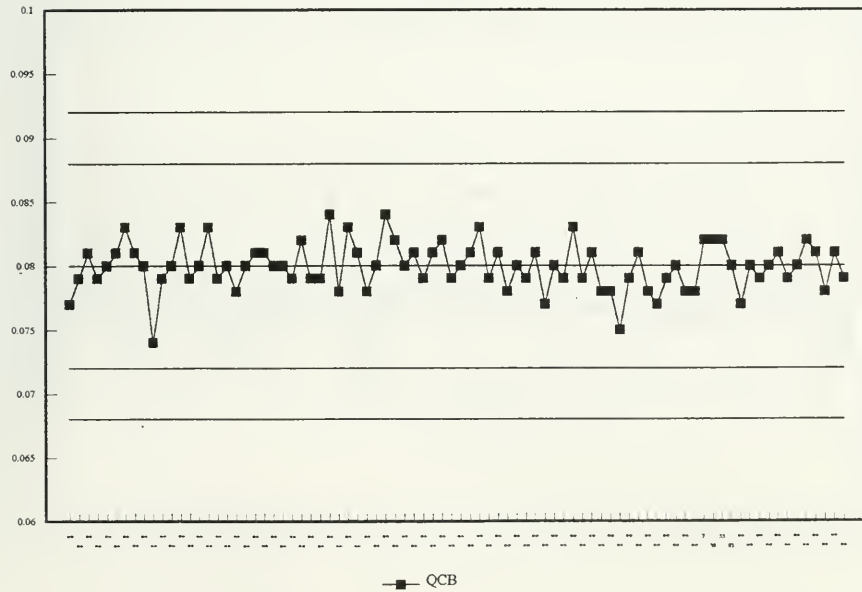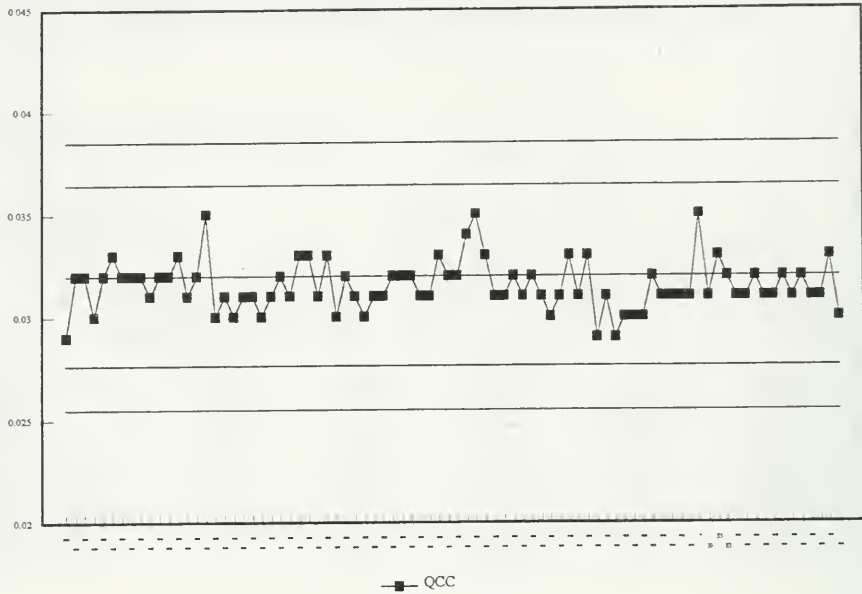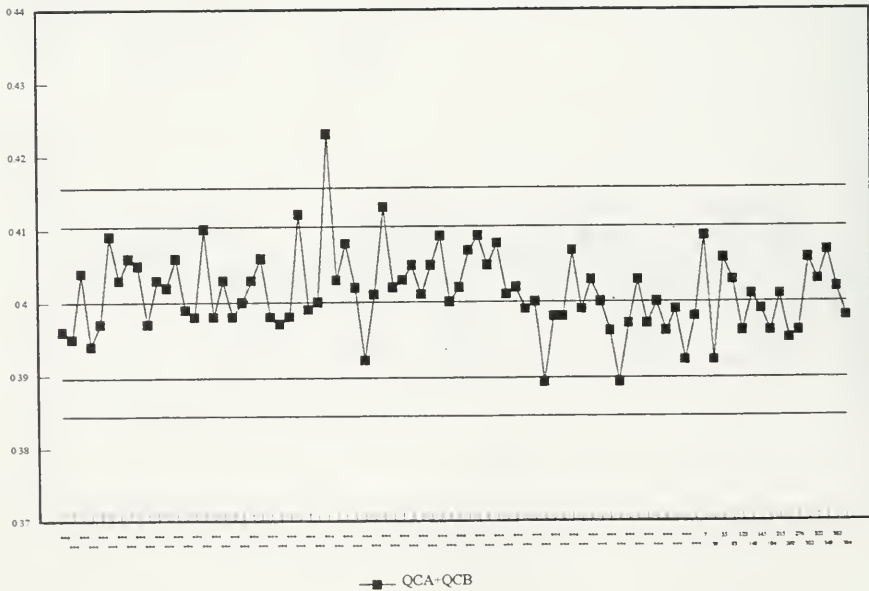
Nitrate, QC Difference

■ QCA-QCB

Nitrate, QCC Sum

■ QCB+QCC

103
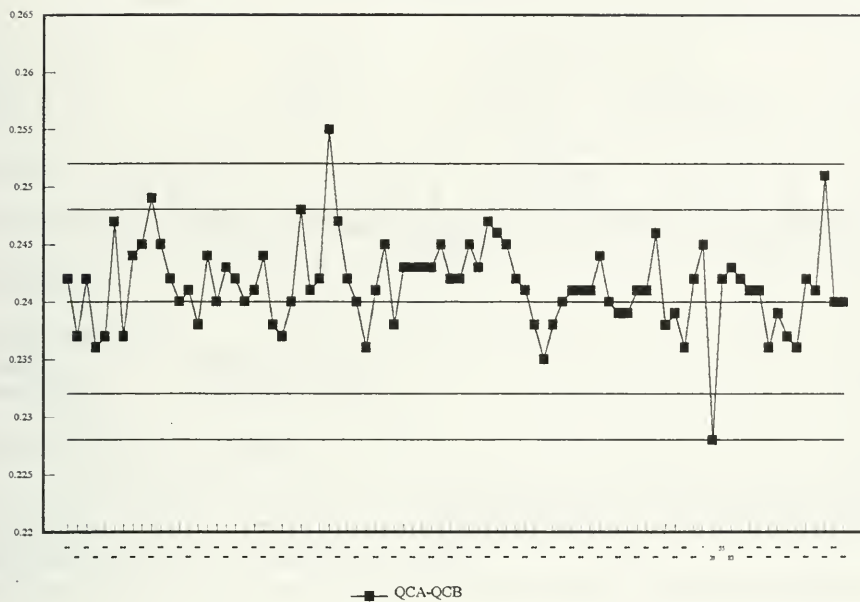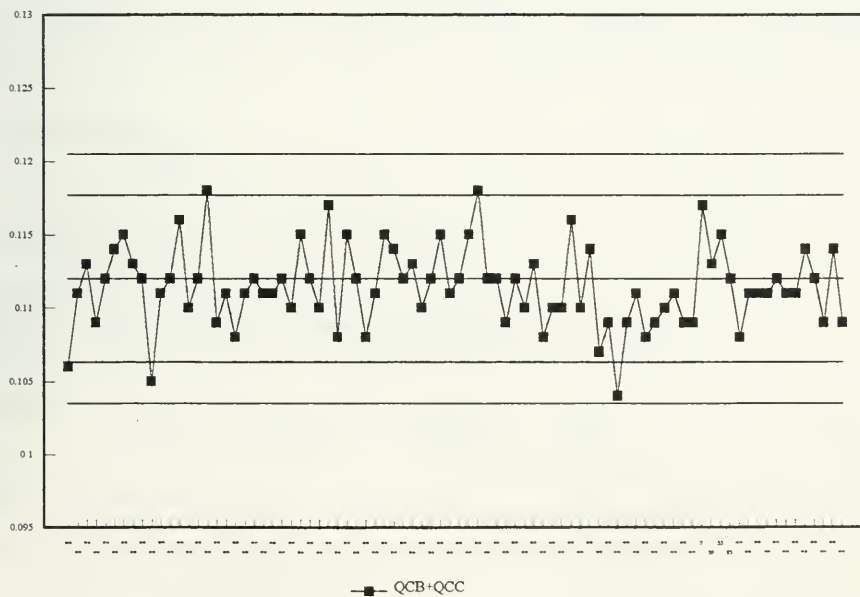
Nitrate, QCC Difference

QCB-QCC
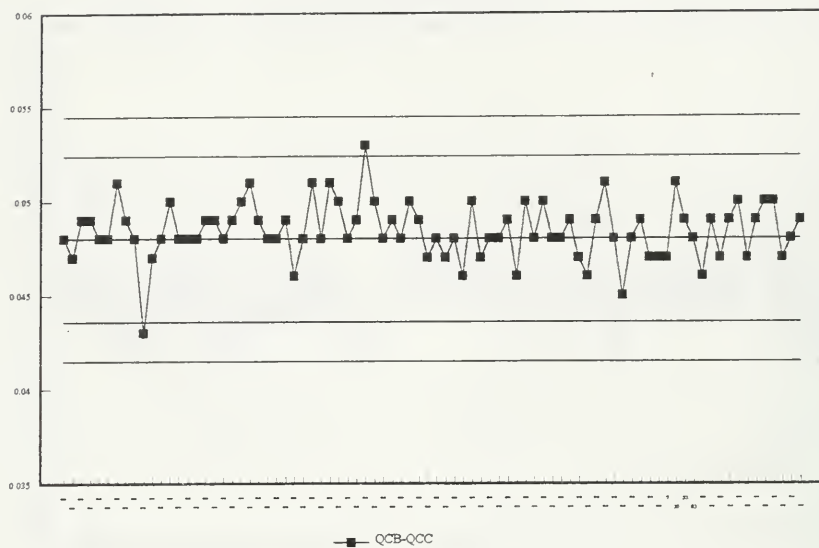
NITRITE

IDENTIFICATION:

LIS Test Name Code:	NNO2FR	Introduced:	1978
Work Station Code:	TBNDNP	Units:	mg/L as N
Method Code:	E6024A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Special Instructions:
Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Nitrite forms a diazotization product with sulphanilamide which is then coupled with N(1-napthyl) ethylenediamine dihydrochloride at pH 1. A light red colour is produced and the absorbance of the solution is measured at 520 nm.
N.B. Ammonia, nitrate plus nitrite and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Automated continuous flow system, Technicon AAII. Colourimetric measurement is through a 5.0 cm. light path at 520 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION:
- Linear
- 7 Standards   0 - 0.10 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: BLK every 10 samples, CHK (100%) every 20 samples
Duplicates: DUP (3 per run, run at beginning)

Reporting: Maximum Significant Figures: 3
                W Value: 0.001   T Value: 0.005

MODIFICATIONS:

1988 - All channels went to microcomputer control with DCI software.

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 0.10 mg/L as N

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	84	0.080	0.0799	−0.0001	0.00075
QCB:	84	0.020	0.0199	−0.0002	0.00035
QCC:	84	0.008	0.0080	0.0000	0.00015
QCA+QCB:	84	0.100	0.0998	−0.0002	0.00087
QCA−QCB:	84	0.060	0.0601	0.0001	0.00078
QCB+QCC:	84	0.028	0.0279	−0.0001	0.00039
QCB−QCC:	84	0.012	0.0118	−0.0002	0.00038

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.00058$$
$$Sw\ (B-C) = \quad 0.00031$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
231	0.00 − 0.02	0.003	0.0004
7	0.02 − 0.05	0.036	0.0005
1	0.05 − 0.10	0.071	0.0000

Nitrite, QCA
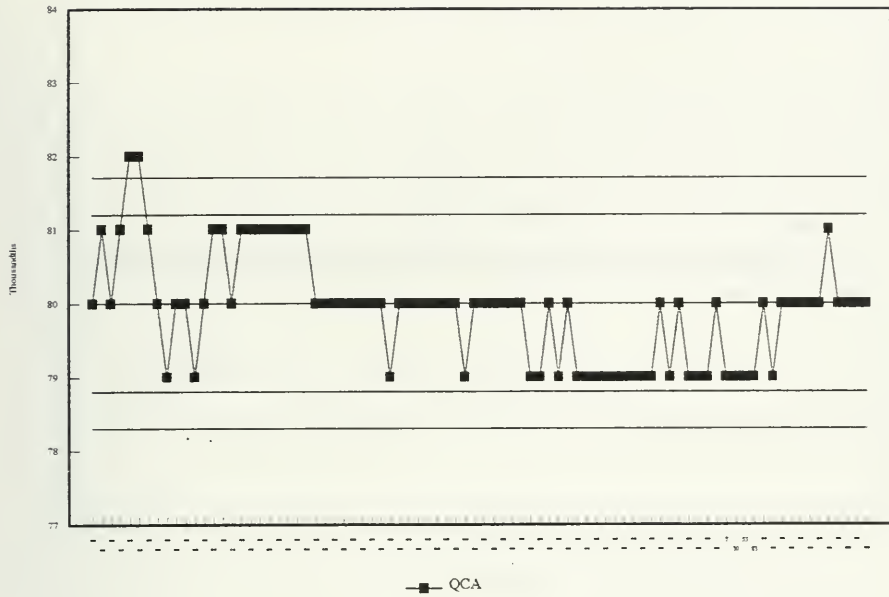
Nitrite, QCB

Nitrite, QCC

QCC

Nitrite, QC Sum

QCA+QCB

108
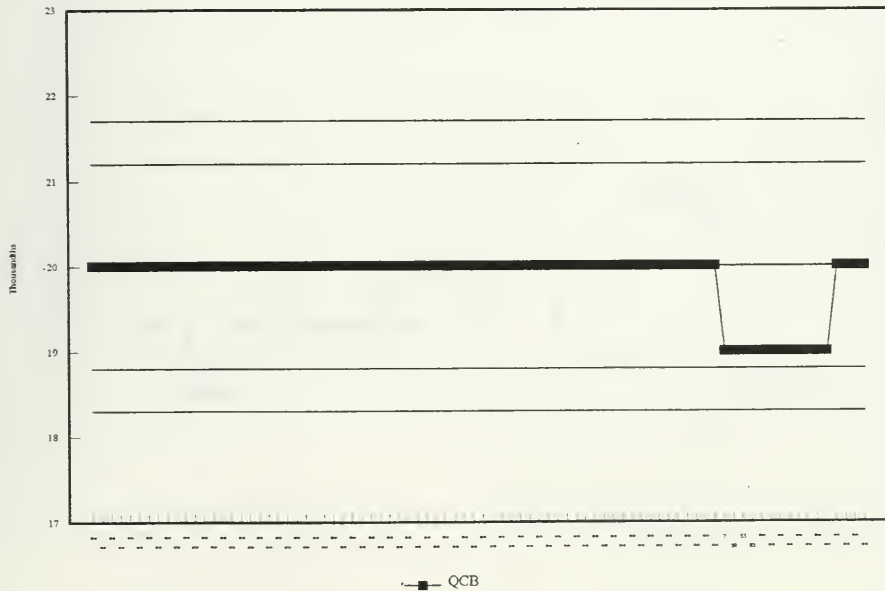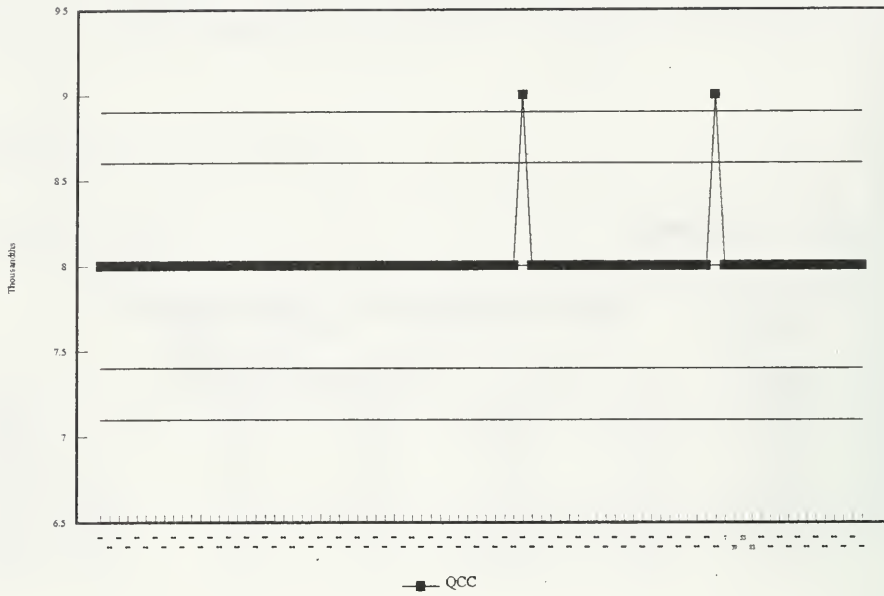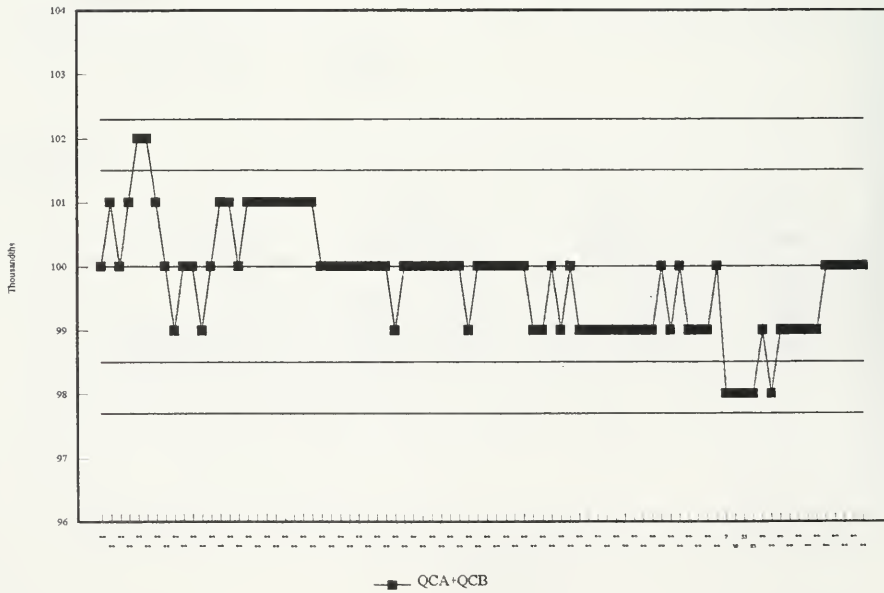
Nitrite, QC Difference
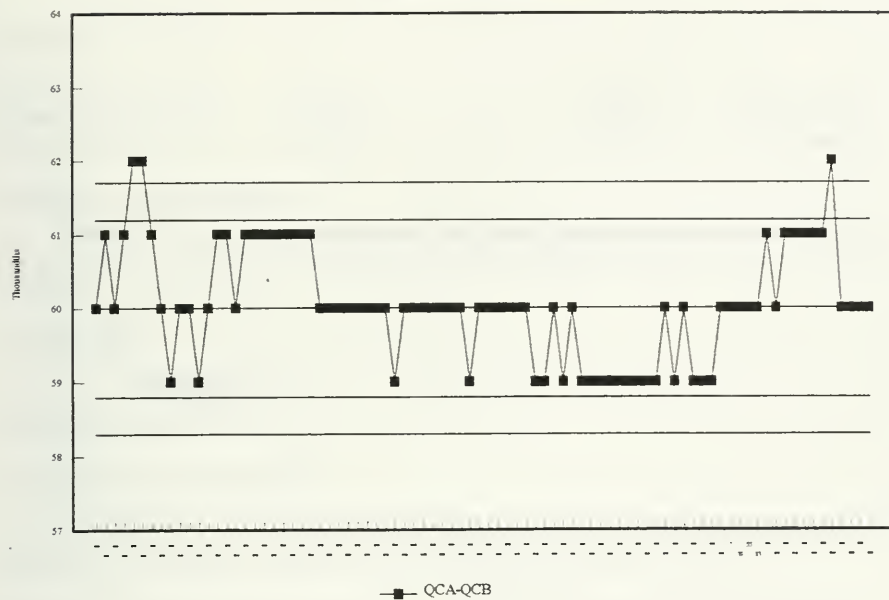
Nitrite, QCC Sum
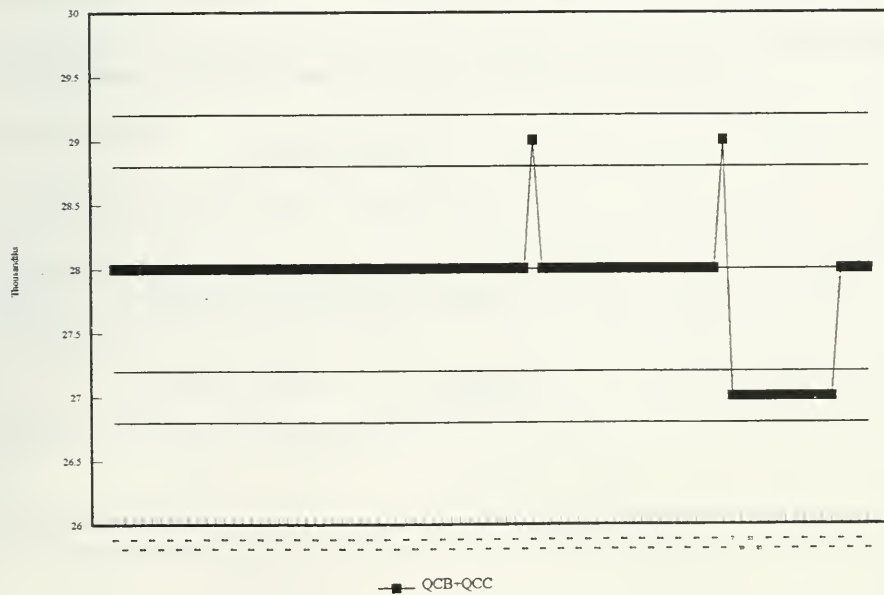
Nitrite, QCC Difference

Legend: ■ QCB-QCC
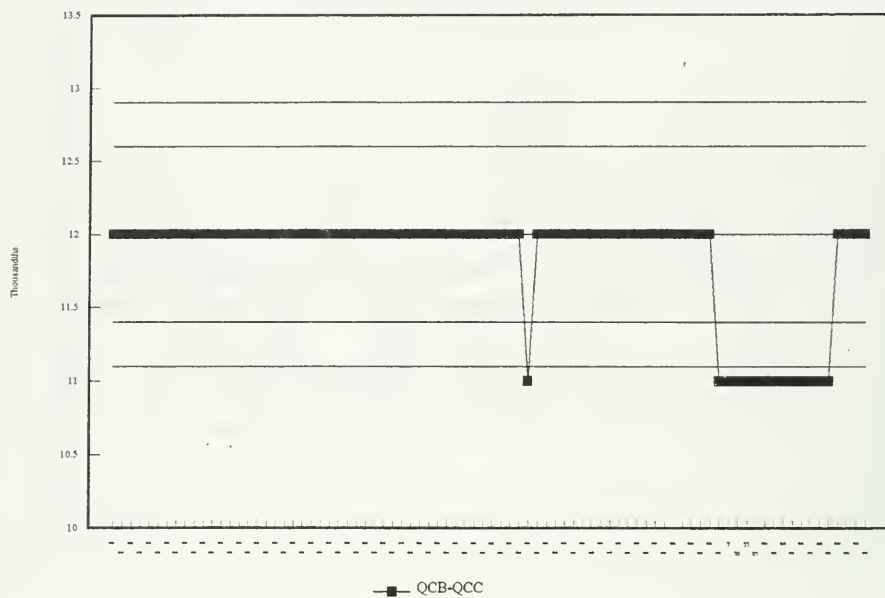
pH

IDENTIFICATION:

LIS Test Name Code:	PH	Introduced:	1978
Work Station Code:	TBCAP	Units:	Dimensionless
Method Code:	E6003A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface waters, drinking water, ground water, sewage effluents, industrial wastes and precipitation samples.

SAMPLING:

Container: Glass or PET jars
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample using a hydrogen ion sensitive glass combination electrode. The meter is calibrated in pH units against buffers of known pH.

NOTE: Total fixed endpoint alkalinity and conductivity are determined simultaneously.

INSTRUMENTATION:

Auto-Titration System, Radiometer, consisting of ABU80 Auto-Burette, SAC80 Multisampler, TTT85 Titrator, CDM83 Conductivity Meter and PRS12 Alpha Printer.

CALIBRATION: - Two point calibration
                     - Buffers 6.86 and 4.01

CONTROLS AND QUALITY ASSURANCE:

Calibration: QCA, QCB
Drift: Buffers are re-analyzed periodically
Duplicates: DUP (1 every 10 samples)

              CAEAL Certified, LRTAP participant

Reporting: Maximum Decimal Places: 1
              W Value: N/A   T Value: N/A

MODIFICATIONS:
1986-Instrumentation changed from Fisher pH Meter to present system.

Quality Control Data from January 1 to December 31, 1993

Analytical Range – 0 to 14

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	182	9.18	9.188	0.008	0.0137
QCB:	182	4.01	4.006	−0.004	0.0082
QCA+QCB:	182	13.19	13.194	0.004	0.0152
QCA−QCB:	182	5.17	5.182	0.012	0.0167

For 1993 Control Charts:

Sw (A−B) =   0.0167

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
10	5.0 – 5.8	5.417	0.0860
93	5.8 – 7.0	6.549	0.0642
402	7.0 – 9.0	7.673	0.0507

pH, QCA

QCA

pH, QCB

QCB

113

pH, QC Sum

—■— QCA+QCB

pH, QC Difference

—■— QCA-QCB
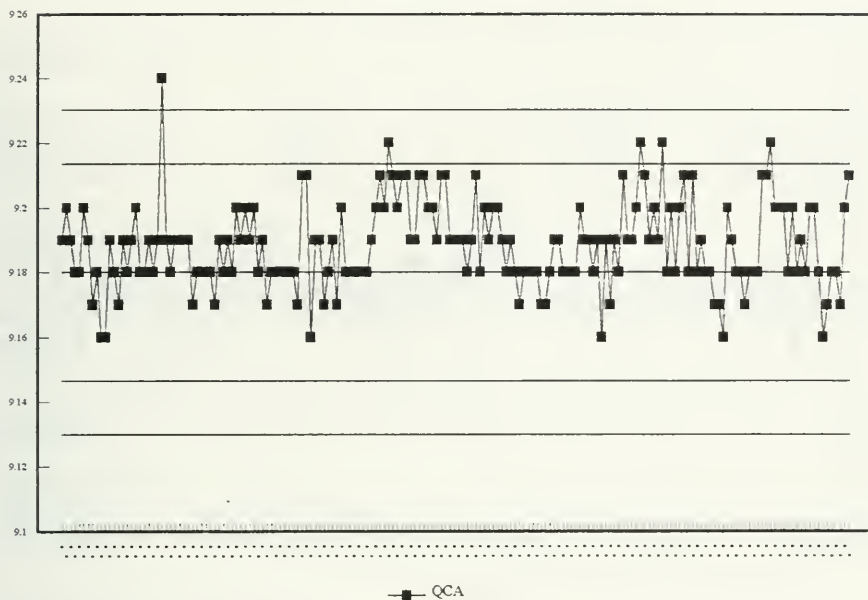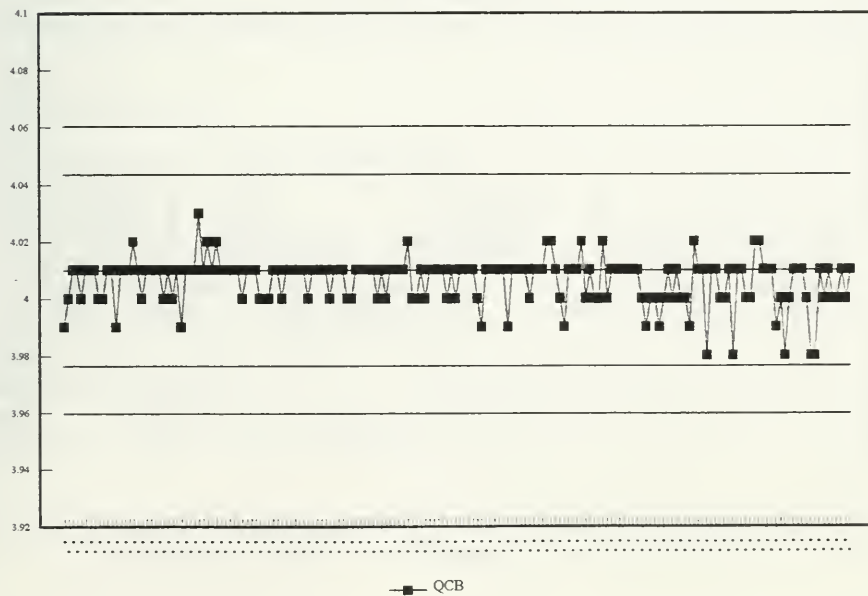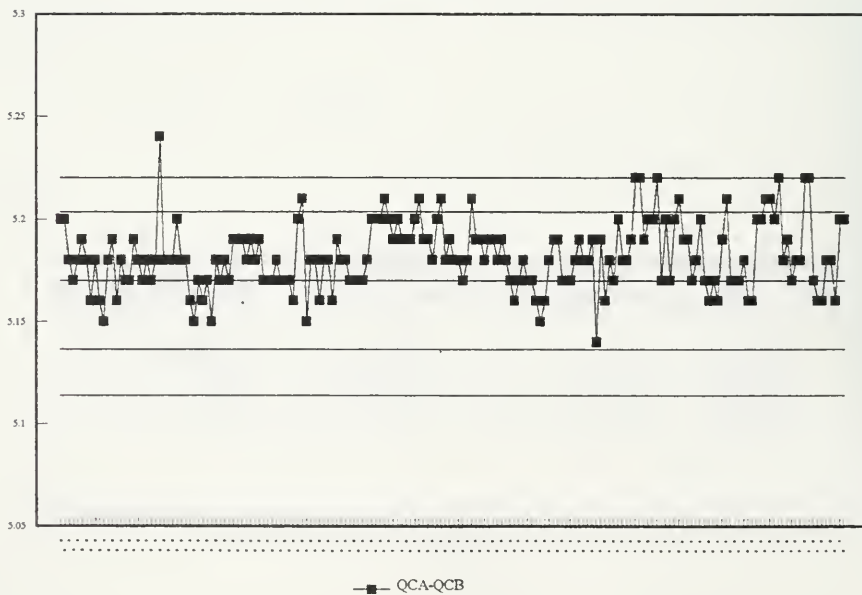
pH - pH PROBE

IDENTIFICATION:

LIS Test Name Code:	PHPROB	Introduced:	May, 1977
Work Station Code:	TBPH	Units:	Dimensionless
Method Code:	E6009A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface waters, drinking water, ground water, sewage effluents, industrial wastes and precipitation samples.

SAMPLING:

Special Instructions:
Container: Glass or PET jars
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

pH is measured directly on a stirred sample using a hydrogen ion sensitive glass combination electrode. The meter is calibrated in pH units against buffers of known pH.

INSTRUMENTATION:

Fisher Accumet 925 pH Meter, magnetic stirrer.

CALIBRATION:
                - Two point calibration
                - Buffers 6.86 and 4.01

CONTROLS AND QUALITY ASSURANCE:

Calibration: QCA, QCB
Drift: Buffers are re-analyzed periodically
Duplicates: DUP (1 every 10 samples)

Reporting: Maximum Significant Figures: 2
             W Value: N/A   T Value: N/A

Analytical Range — 0 — 14

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	167	9.18	9.180	0.000	0.0225
QCB:	167	4.01	4.009	−0.001	0.0064
QCA+QCB:	167	13.19	13.188	−0.002	0.0237
QCA−QCB:	167	5.17	5.171	0.001	0.0230

For 1993 Control Charts:

$$Sw (A-B) = \qquad 0.0296$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
14	5.0 — 5.8	5.543	0.0181
73	5.8 — 7.0	6.575	0.0163
223	7.0 — 9.0	7.486	0.0218

pH Probe, QCA

QCA

pH Probe, QCB

QCB

pH Probe, QC Sum

QCA+QCB
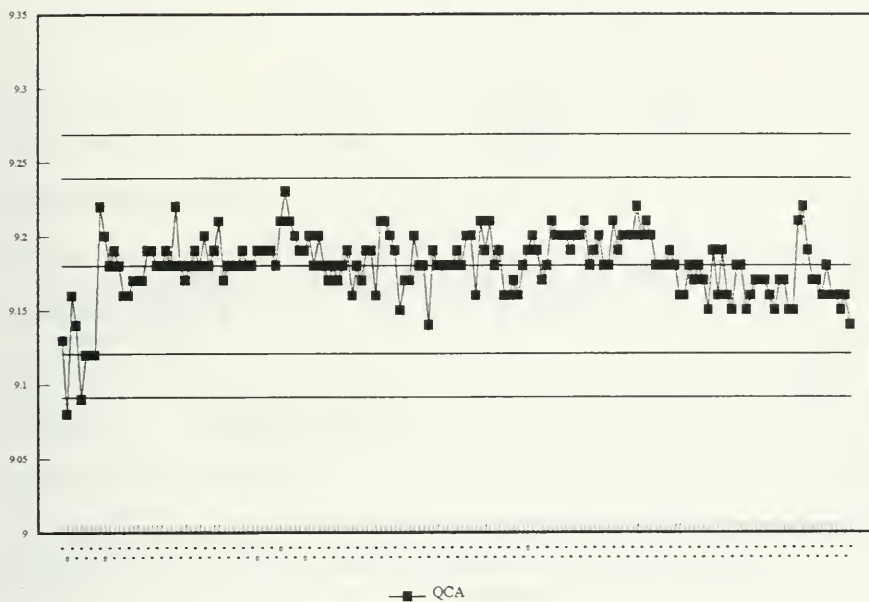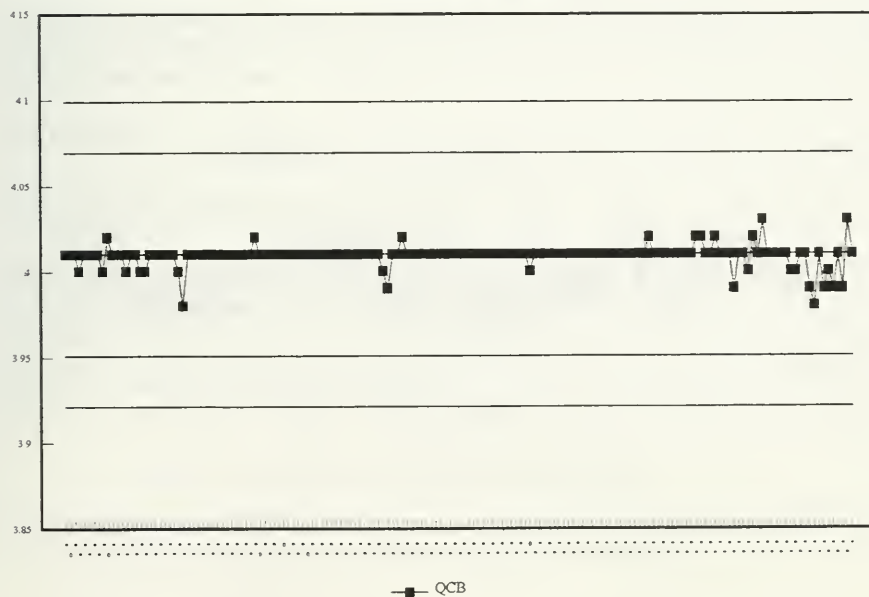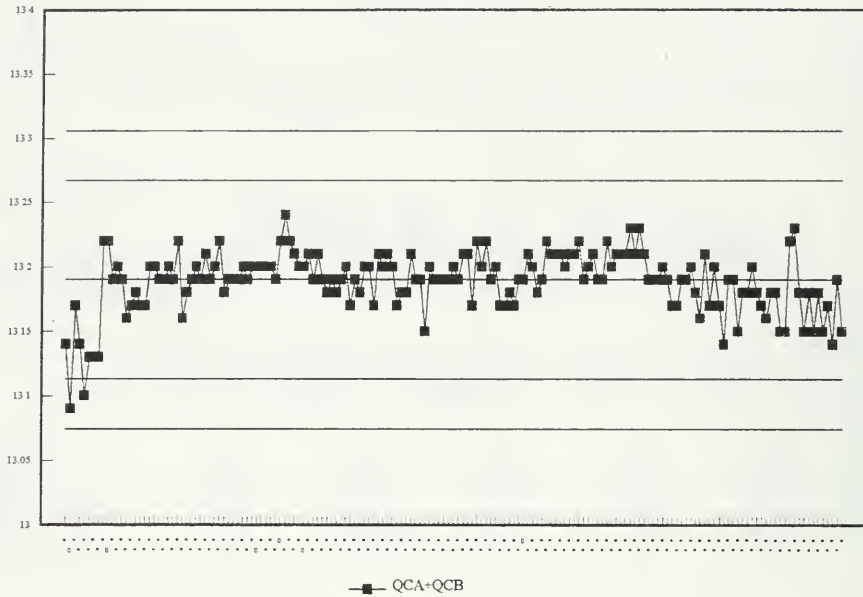
pH Probe, QC Difference

QCA-QCB

118
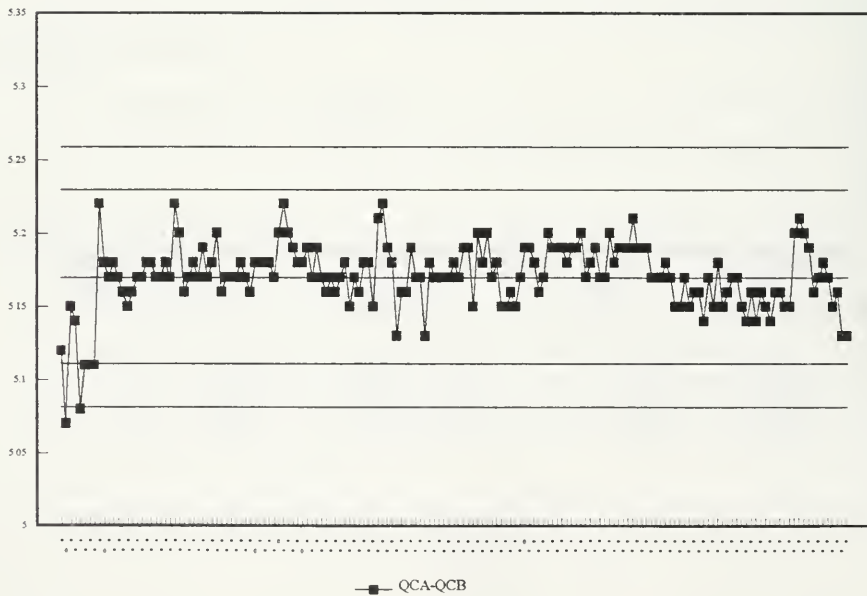
PHOSPHORUS-REACTIVE ORTHOPHOSPHATE

IDENTIFICATION:

LIS Test Name Code:	PPO4FR	Introduced:	1978
Work Station Code:	TBNDNP	Units:	mg/L as P
Method Code:	E6024A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Special Instructions:
Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:

Orthophosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
N.B. Ammonia, nitrate plus nitrite, and nitrite are determined simultaneously.

INSTRUMENTATION:

Automated continuous flow system, Technicon AAII with 37°C heating bath. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION: - Linear
                    - 7 Standards   0 - 0.10 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: BLK every 10 samples, CHK (100%) every 20 samples
Duplicates: DUP (3 per run, run at beginning)

Reporting: Maximum Significant Figures: 3
               W Value: 0.001   T Value: 0.005

               CAEAL Accredited and QM Blind Audit participant.

MODIFICATIONS:
1988 - All channels went to microcomputer control with DCI software.

PHOSPHORUS—REACTIVE ORTHOPHOSPHATE

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 0.10 mg/L as P

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	84	0.080	0.079	−0.001	0.0015
QCB:	84	0.020	0.019	−0.001	0.0008
QCC:	84	0.008	0.007	−0.001	0.0007
QCA+QCB:	84	0.100	0.098	−0.002	0.0021
QCA−QCB:	84	0.060	0.060	0.000	0.0012
QCB+QCC:	84	0.028	0.026	−0.002	0.0013
QCB−QCC:	84	0.012	0.012	0.000	0.0006

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.0012$$
$$Sw\ (B-C) = \quad 0.0012$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
187	0.00 − 0.02	0.004	0.0008
28	0.02 − 0.05	0.031	0.0020
18	0.05 − 0.10	0.069	0.0020

Phosphates, QCA

QCA

Phosphates, QCB

QCB

121
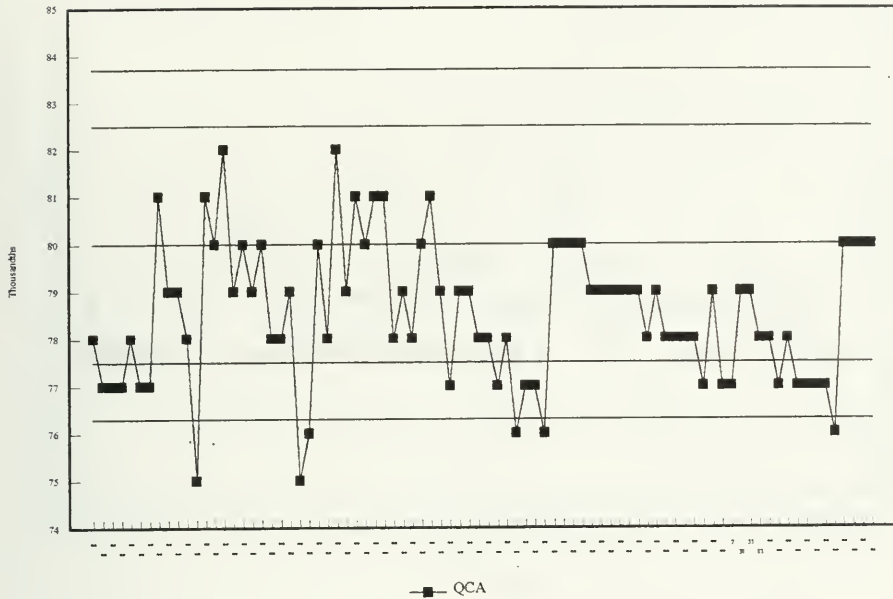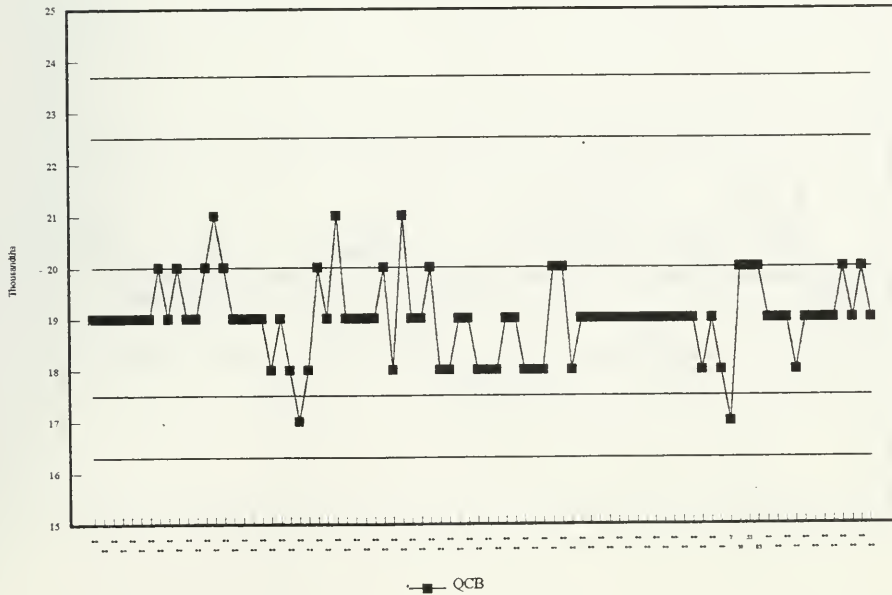
Phosphates, QCC

QCC

Phosphates, QC Sum

QCA+QCB
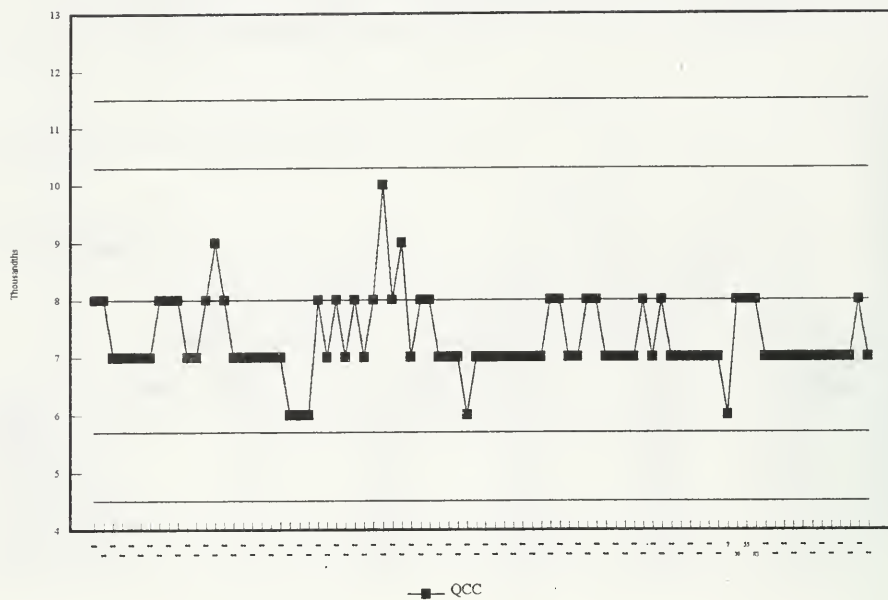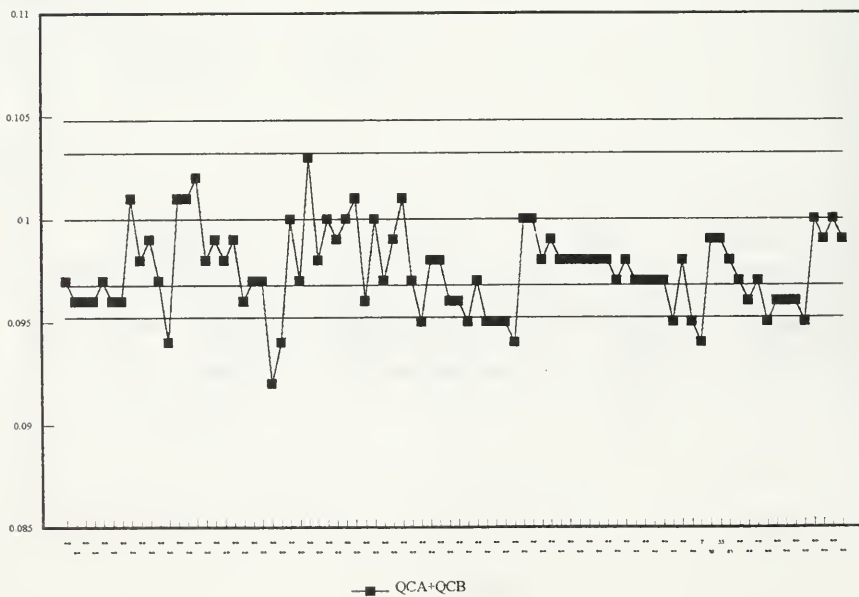
Phosphates, QC Difference

QCA-QCB
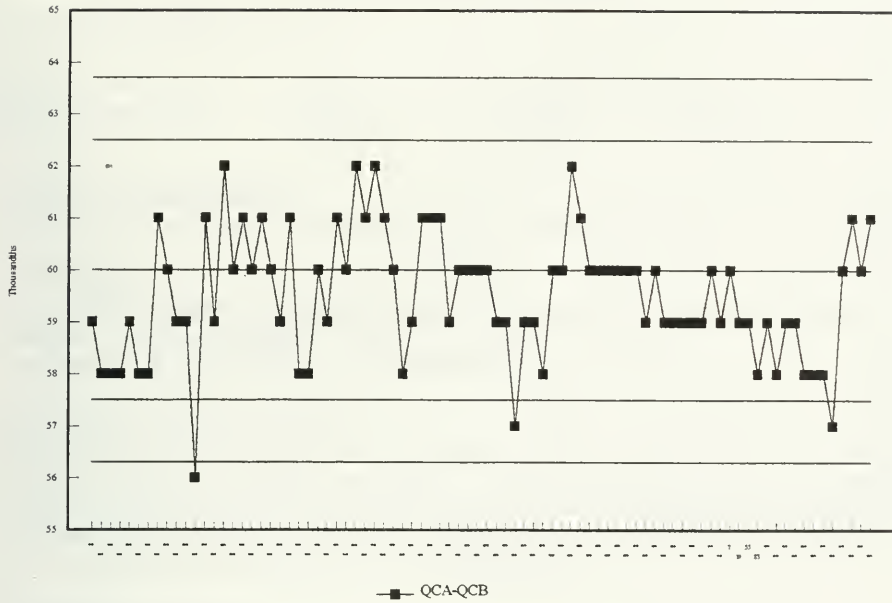
Phosphates, QCC Sum

QCB+QCC

123
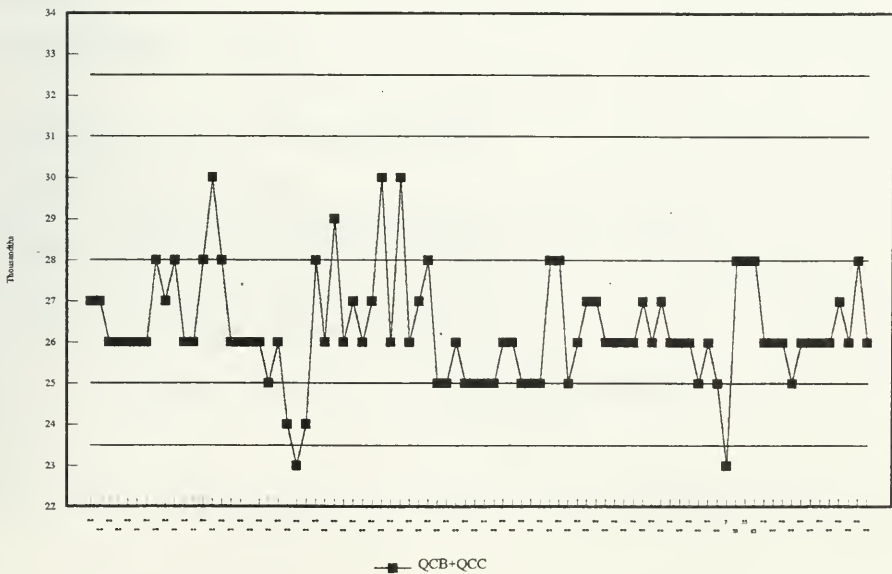
Phosphates, QCC Difference
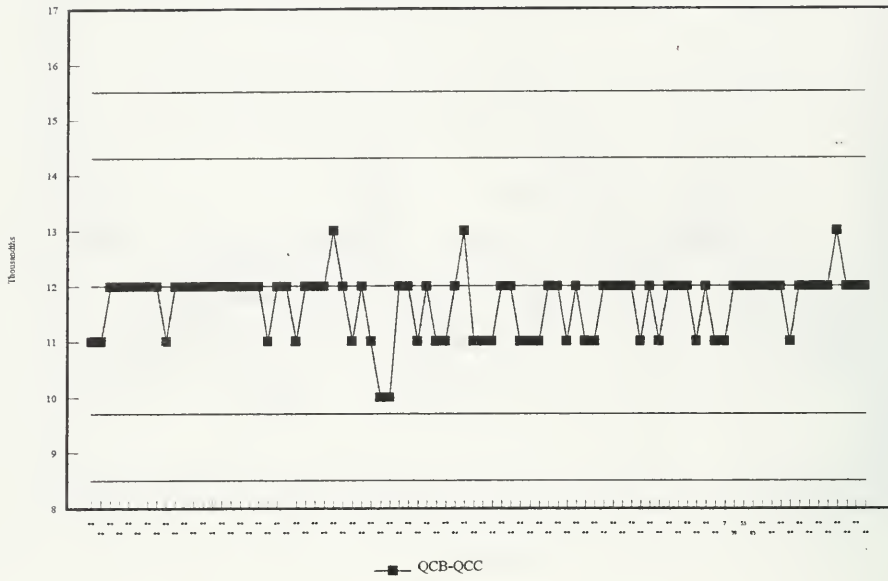
PHOSPHORUS - TOTAL

IDENTIFICATION:

LIS Test Name Code:	PPUT	Introduced:	1978
Work Station Code:	TBTNTP	Units:	mg/L as P
Method Code:	E6026A	Section:	Water Quality

SAMPLE TYPE/MATRIX:
Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:
Samples are digested stepwise in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters set at 200°C and 360°C. The orthophosphate content in the digestate is determined by the formation of a reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
N.B. Total Kjeldahl Nitrogen is determined simultaneously.

INSTRUMENTATION:
Automated continuous flow system, Technicon AAII. Colourimetric measurement is through a 5.0 cm. light path at 880 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION: - Linear
                                 - 7 Standards   0 - 0.16 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB
Drift: BLK every 10 samples, CHK (100%) every 20 samples
Duplicates: DUP (3 per run, run at beginning)

Reporting: Maximum Significant Figures: 3
              W Value: 0.001 T Value: 0.005

              CAEAL Accredited and QM Blind Audit participant

MODIFICATIONS:

February 1989 - Both channels went to microcomputer control with DCI software.

TOTAL PHOSPHORUS

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 0.16 mg/L as P

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	96	0.15	0.150	0.000	0.0016
QCB:	96	0.05	0.050	0.000	0.0011
QCA+QCB:	96	0.20	0.201	0.001	0.0021
QCA−QCB:	96	0.10	0.100	0.000	0.0016

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.0012$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
172	0.000 − 0.032	0.011	0.0016
47	0.032 − 0.080	0.055	0.0031
31	0.080 − 0.160	0.111	0.0047

Phosphorus-Total, QCA

Phosphorus-Total, QCA

Phosphorus-Total, QCB
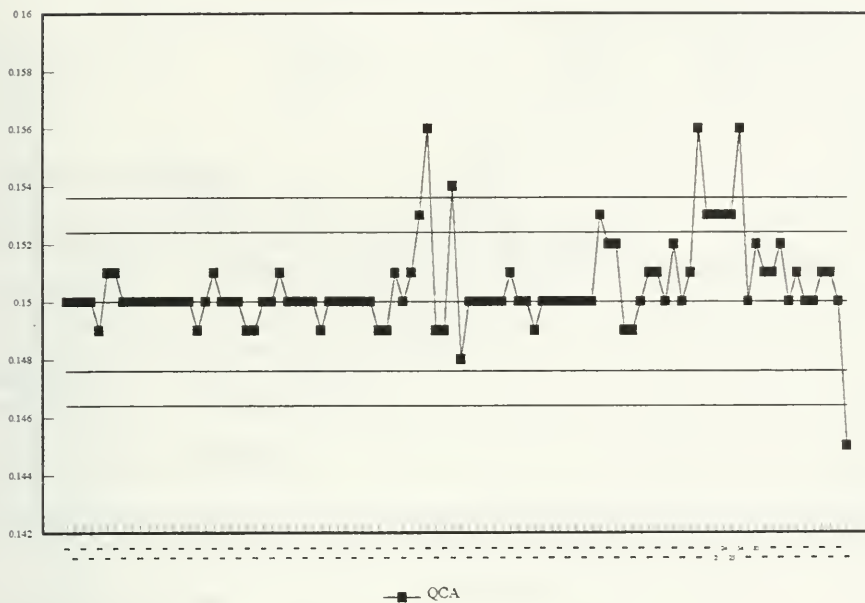
Phosphorus-Total, QCB
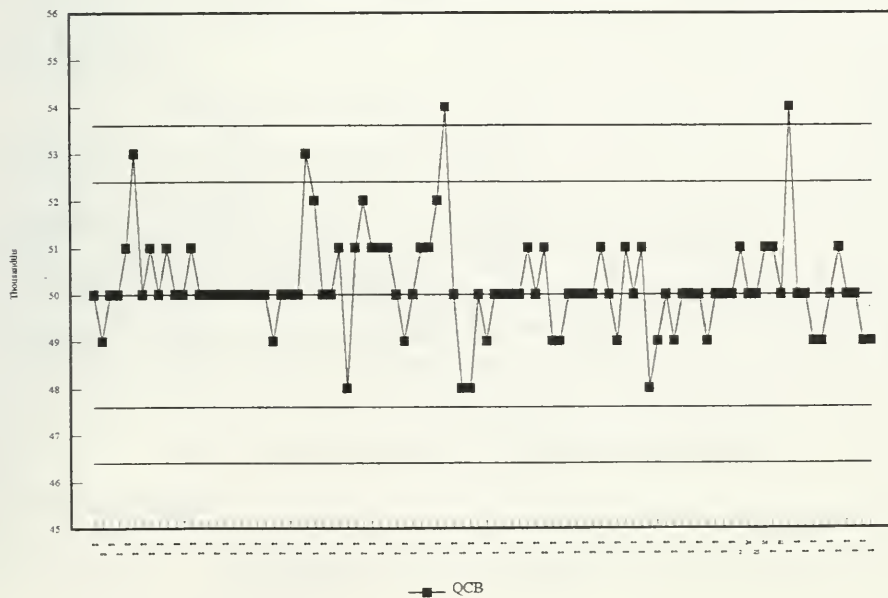
Phosphorus-Total, QC Sum

QCA+QCB
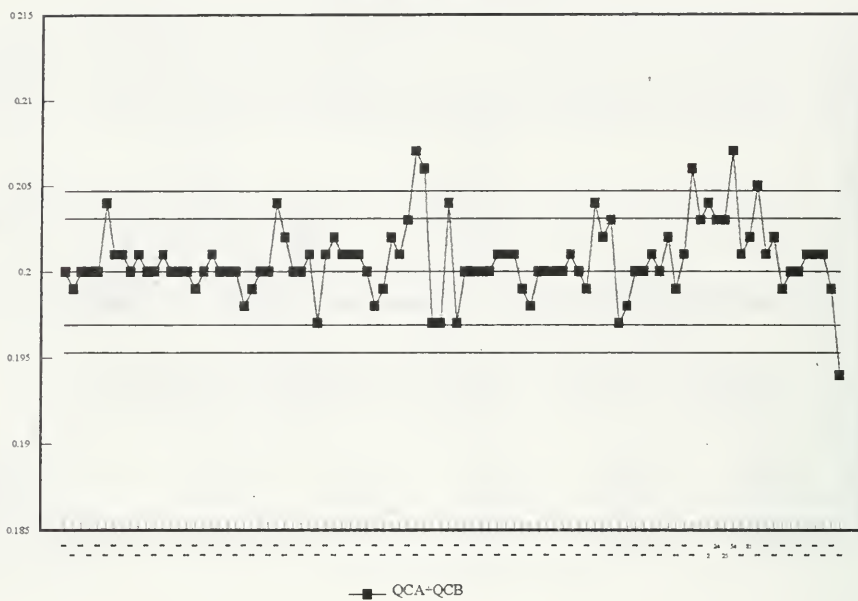
Phosphorus-Total, QC Difference

QCA-QCB

128
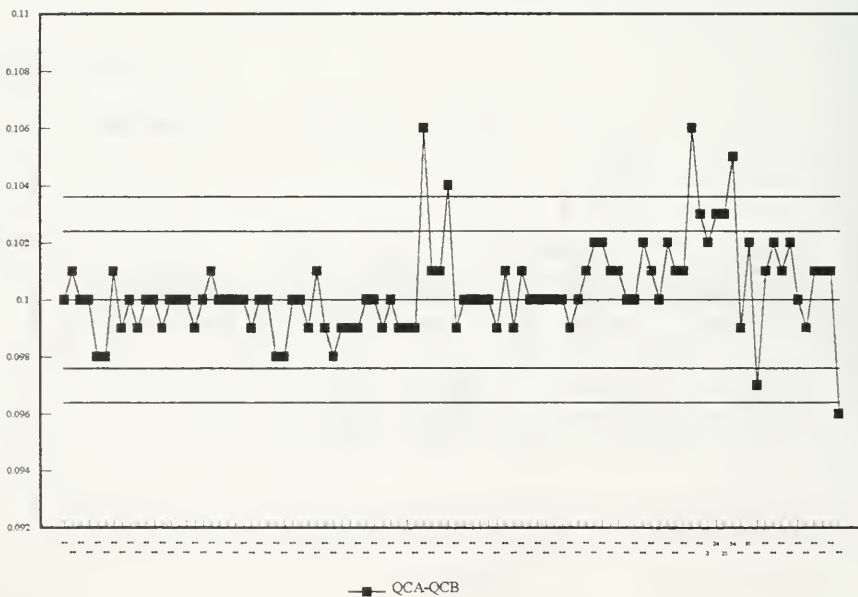
SILICON - MOLYBDATE REACTIVE SILICATES

IDENTIFICATION:

LIS Test Name Code: SIO3UR   Introduced: January 1978
Work Station Code: TBSIO3UR   Units: mg/L as Si
Method Code: E6025A   Section: Water Quality

SAMPLE TYPE/MATRIX:

Surface waters, domestic waters, leachates and industrial effluents.

SAMPLING:

Special Instructions: Do not use glass containers.
Container: PET or Nalgene
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

Reactive silicates are determined by the formation of a reduced molybdo-silicate complex at pH 1.2. Ascorbic acid is the reducing agent. Oxalic acid suppresses phosphate interference.

INSTRUMENTATION:

Technicon Auto-Analyzer II continuous flow system with colourimetric measurement through a 50 mm light path at 660 nm.

CALIBRATION:
- Linear
- 8 Standards, 0 - 3.0 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB
Drift: BLK (every 10 samples); SENS.CHK (2 every 20 samples)
Duplicates: DUP (every 10 samples)

Reporting :   Maximum Significant Figures: 2
               W Value: .02   T Value: .10

               CAEAL Accredited, LRTAP and QM Blind Audit participant

MODIFICATIONS:

January 1990 -   The reporting procedure was changed to include the range 0.02 mg/L to 0.1 mg/L.

SILICON − MOLYBDATE REACTIVE SILICATES

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 3.0 mg/L as Si

Calibration Control:

|	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	28	1.4	1.3738	−0.026	0.0167
QCB:	28	0.2	0.1925	−0.008	0.0070
QCA+QCB:	28	1.6	1.5663	−0.034	0.0180
QCA−QCB:	28	1.2	1.1814	−0.019	0.0183

For 1993 Control Charts:

$$Sw (A-B) = \quad 0.0145$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
5	0 − 0.6	0.260	0.0049
30	0.6 − 1.5	1.165	0.0201
21	1.5 − 3.0	1.842	0.0261

Silicates, QCA

— QCA
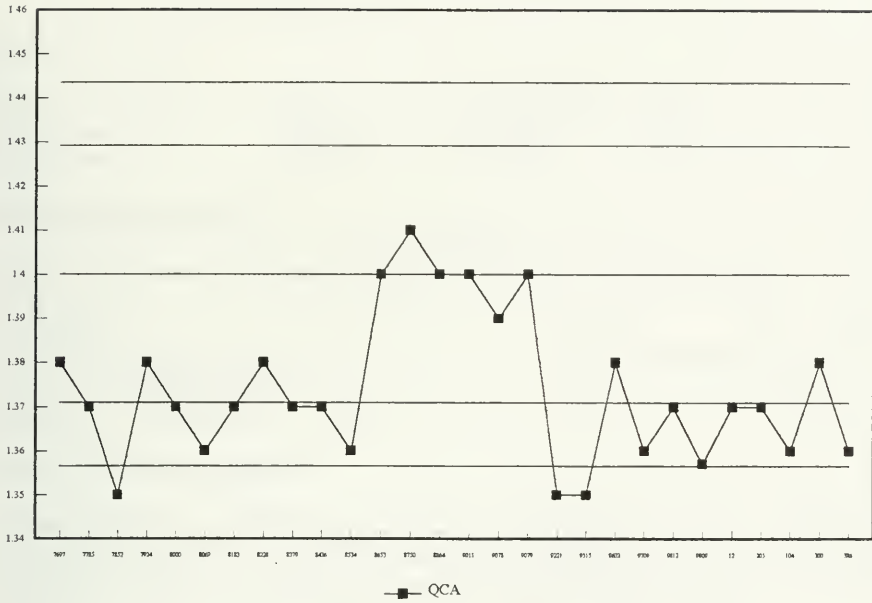
Silicates, QCB

— QCB

Silicates, QC Sum

QCA+QCB
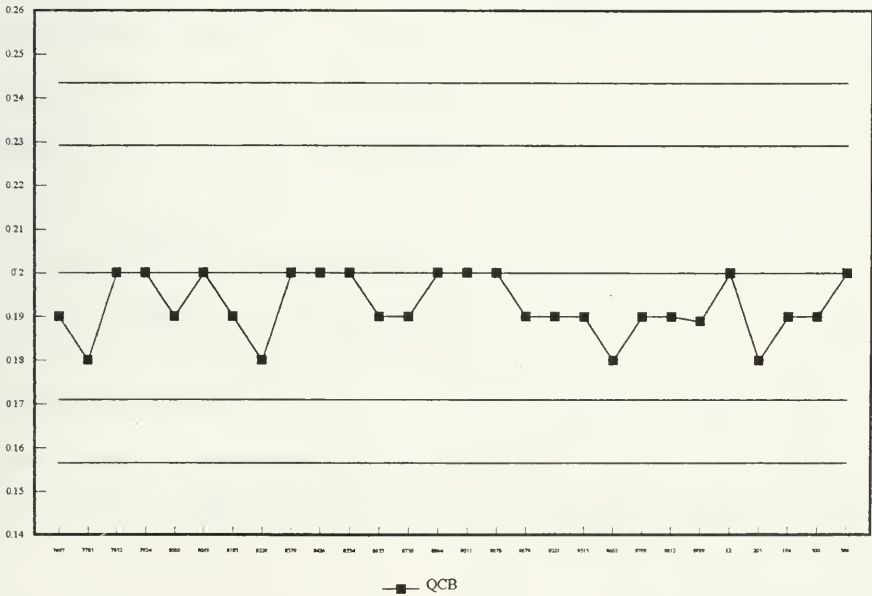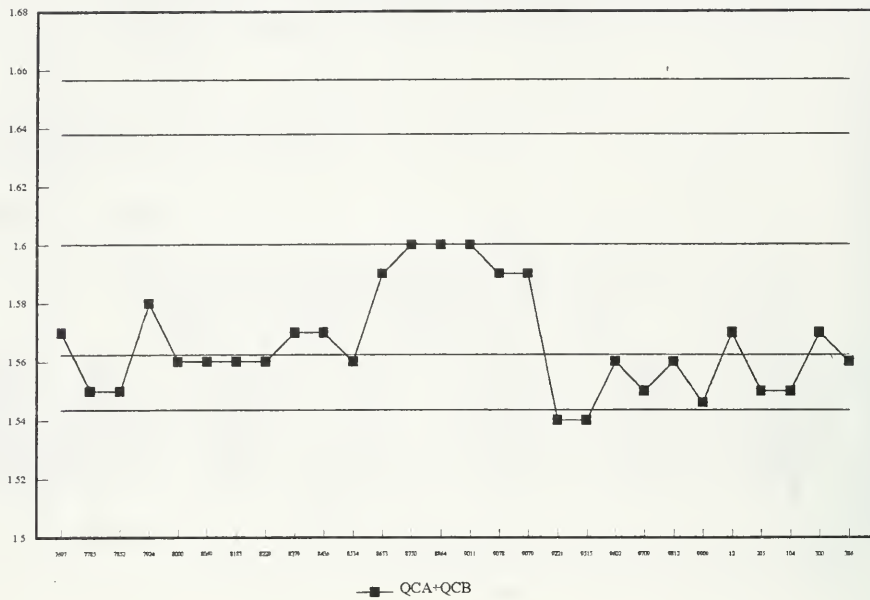
Silicates, QC Difference

QCA-QCB
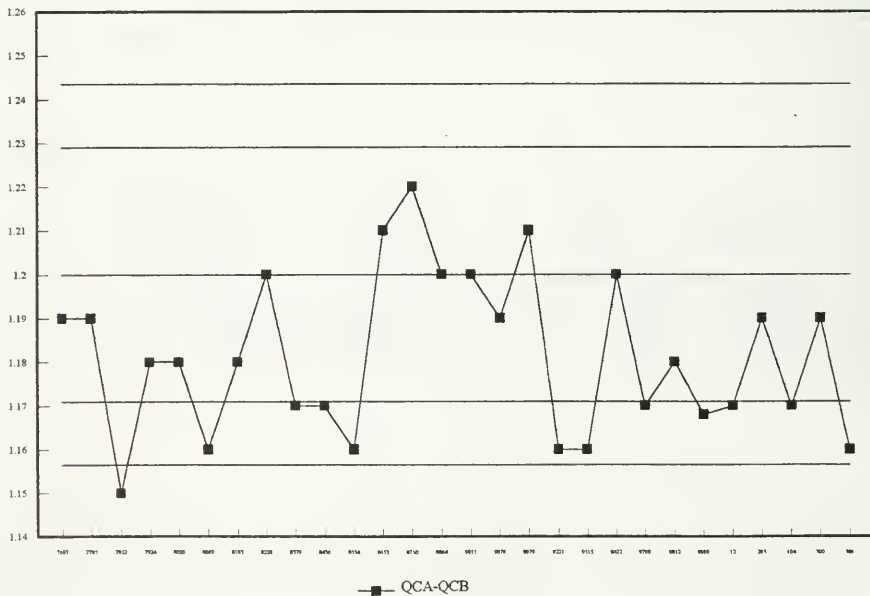
SOLIDS - DISSOLVED

IDENTIFICATION:

LIS Test Name Code:	RSF	Introduced:	1980
Work Station Code:	TBSOLIDS	Units:	mg/L
Method Code:	E6030A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Sewages, surface waters, precipitation, industrial effluents and landfill leachates.

SAMPLING:

Container:	PET or glass
Preservative:	Refrigerate at 4°C

ANALYTICAL PROCEDURE:

A volume of sample is vacuum filtered through a pre-washed glass fibre filter; a 50 mL aliquot if filtrate is evaporated in a pre-weighed ceramic dish overnight at 103°C ± 2°C. The increase in weight over that of the empty dish represents the dissolved solids. Data collection, calculations and transfer of results are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 place), drying oven, micro-computer system with appropriate software.

CALIBRATION: - Balance internal calibration, tare

CONTROLS AND QUALITY ASSURANCE:

Calibration:	2 S class weights
Drift:	Balance zero
Duplicates:	DUP (1 for every 10 samples)

Reporting:	Maximum Significant Figures: 2
	W Value: 5.0 T Value: 25.0

MODIFICATIONS:

September, 1984 - Commodore computer set up for input.
September, 1988 - Direct Computer Input with Commodore computer.
June, 1989 - Microcomputer and in-house Lotus program replaced Commodore computer.

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 4000.0 mg/L

Calibration Control:

|	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA	19	50.00007	50.00003	−0.00004	0.000029
QCB	19	30	29.99996	−0.00004	0.000019
QCA+QCB	19	80.00007	79.99999	−0.00008	0.000042
QCA−QCB	19	20.00007	20.00006	−0.00001	0.000025

For 1993 Control Charts:

$$Sw (A-B) = \quad 0.000035$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
30	0 – 800	128.4	6.94
4	800 – 2000	1367.5	8.6
2	2000 – 4000	3461	11.51

Solids-Dissolved, QCA
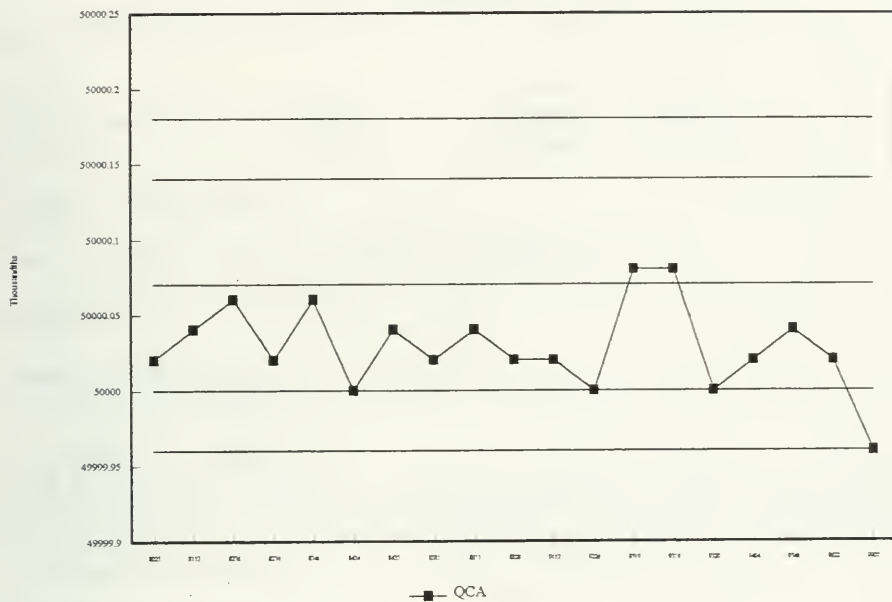
Solids-Dissolved, QCB
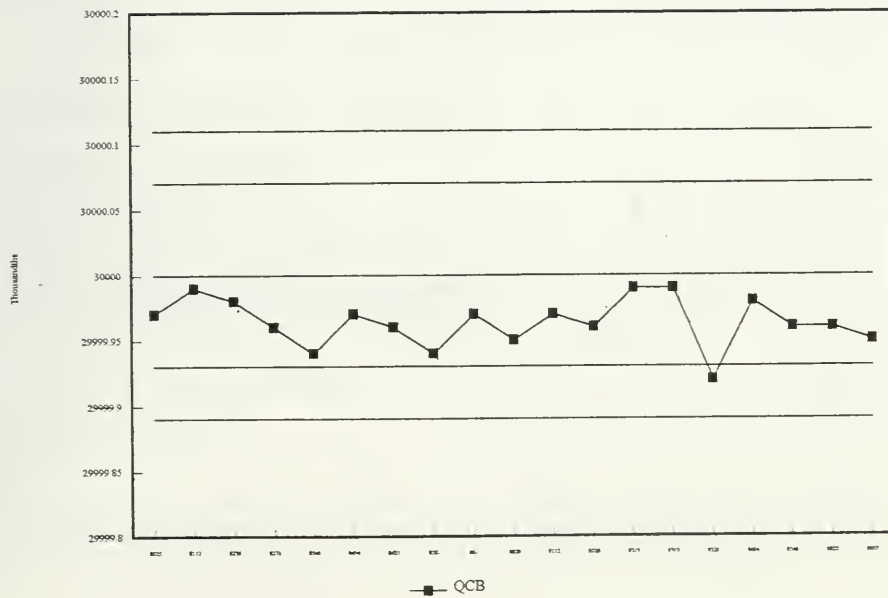
Solid-Dissolved, QC Sum
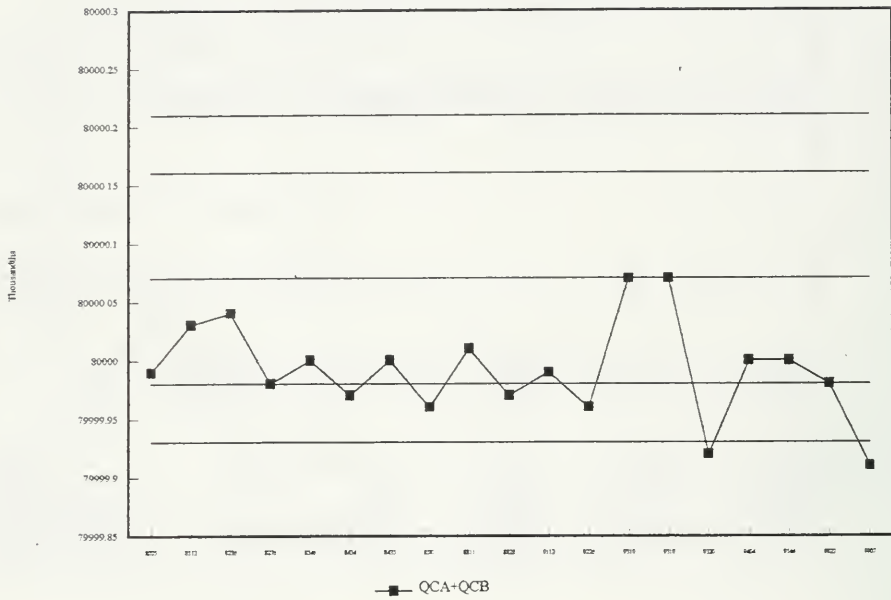
Solids-Dissolved, QC Difference
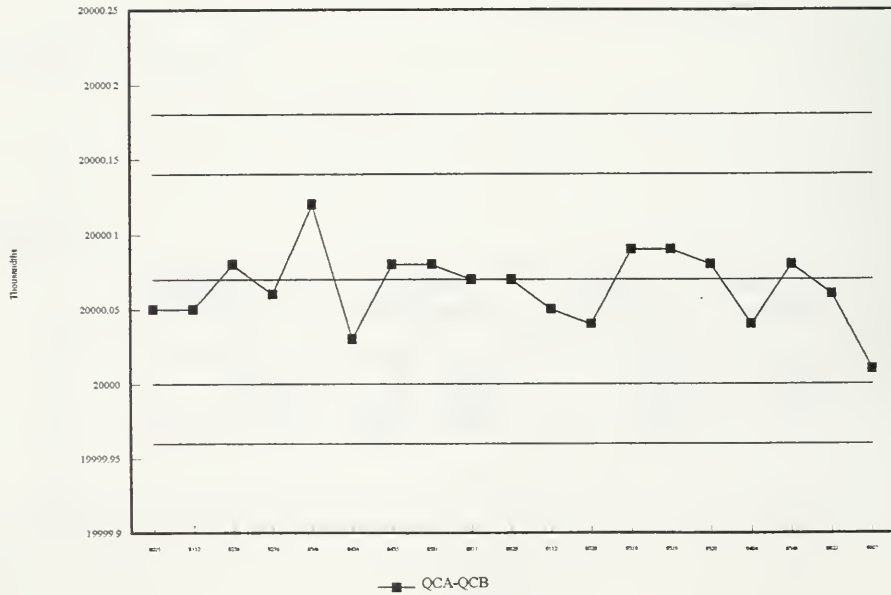
SOLIDS - PARTICULATE

IDENTIFICATION:

LIS Test Name Code:	RSP	Introduced:	1980
Work Station Code:	TBSOLIDS	Units:	mg/L
Method Code:	E6035A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Sewages, surface waters, industrial effluents and landfill leachates.

SAMPLING:

Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:
An aliquot of sample (50-500 mL) is vacuum filtered through a pre-weighed Whatman 934 AH glass fibre filter paper. The residue is dried at 103°C ±2°C and the particulate solids are determined gravimetrically. Data collection, calculations and transfer of results are controlled by a microcomputer system.

INSTRUMENTATION:
Balance (5 place), drying oven, vacuum filtration apparatus, micro-computer system with appropriate software.

CALIBRATION: - Balance internal calibration, tare

CONTROLS AND QUALITY ASSURANCE:

Calibration: 2 S class weights
Recovery: LTB, REC1, REC2
Drift: Balance zero
Duplicates: DUP (1 for every 10 samples)

Reporting: Maximum Significant Figures: 2
              W Value: 1.0   T Value: 5.0

              CAEAL Accredited

MODIFICATIONS:

September, 1984 - Commodore computer set up for input.
September, 1988 - Direct Computer Input with Commodore computer.
June, 1989 - In-house Lotus program replaced Commodore.

SOLIDS – PARTICULATE

Quality Control Data from January 1 to December 31, 1993

Analytical Range – to 200.0 mg/L

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	125	0.49992	0.49991	−0.00001	0.000017
QCB:	125	0.04994	0.04994	0.00000	0.000019
QCA+QCB:	125	0.54986	0.54986	0.00000	0.000280
QCA−QCB:	125	0.44998	0.44997	−0.00001	0.000023

For 1993 Control Charts:

Sw (A−B) = 0.000025

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
418	0 − 40	8.376	0.5950
95	40 − 100	65.895	1.9057
52	100 − 200	133.500	5.0288

138

Solids-Particulate, QCA

QCA
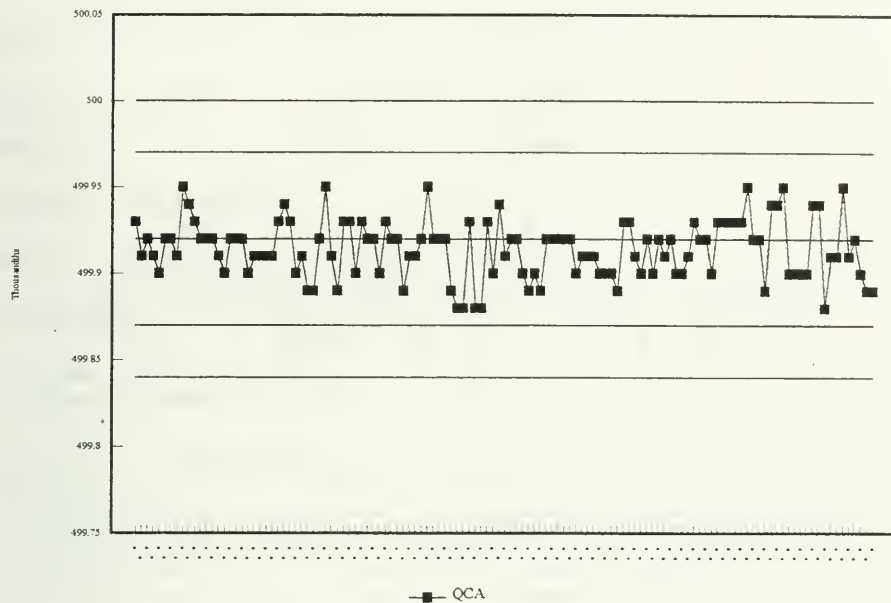
Solids-Particulate, QCB

QCB

139
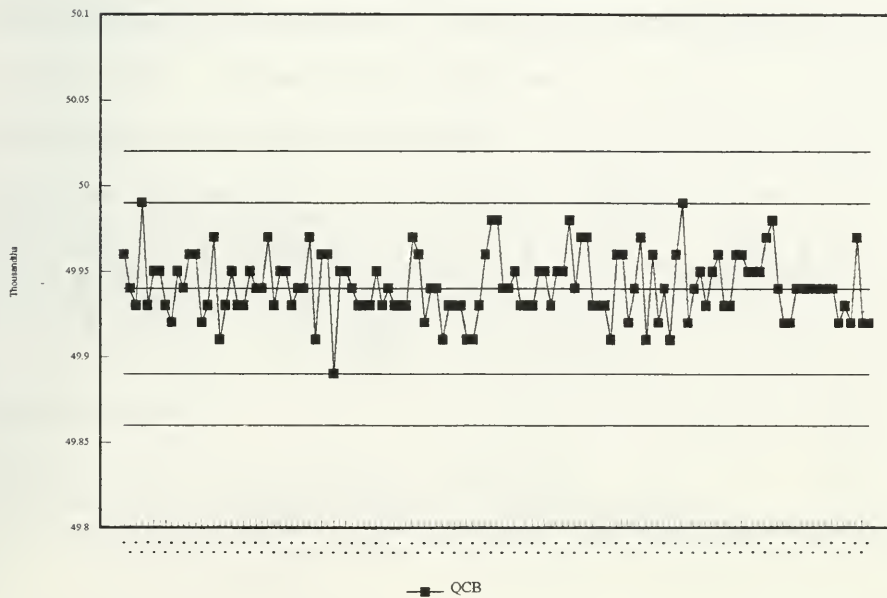
Solids-Particulate, QC Sum

■ QCA-QCB
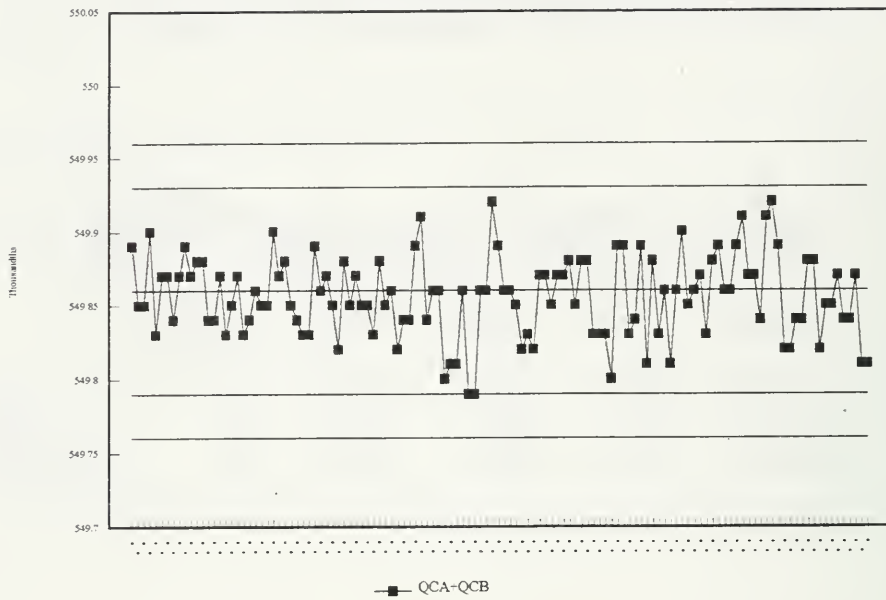
Solids-Particulate, QC Difference

■ QCA-QCB

140
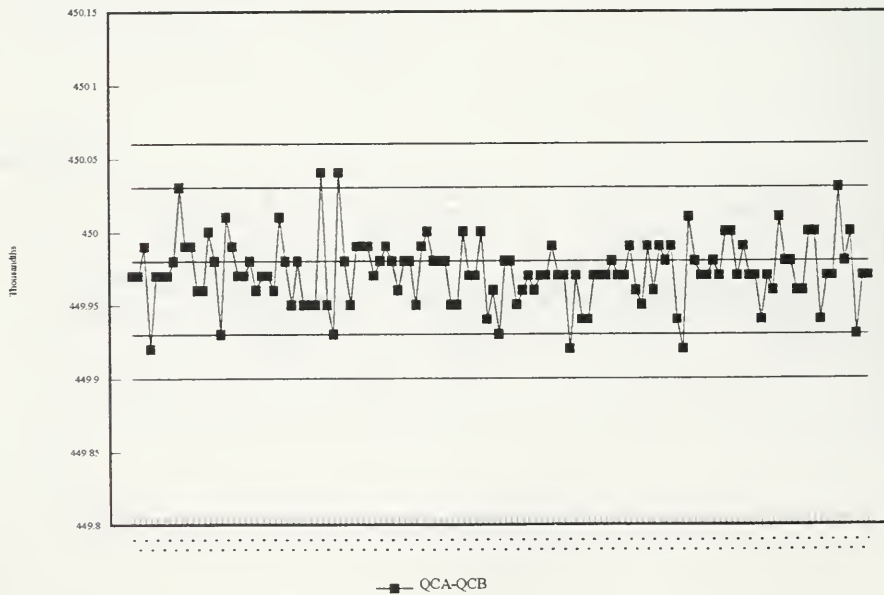
SOLIDS, PARTICULATE - IGNITED

IDENTIFICATION:

LIS Test Name Code:	RSPA, RSPLOI	Introduced:	1980
Work Station Code:	TBRSI	Units:	mg/L
Method Code:	E6029A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Sewages, precipitation, industrial effluents and landfill leachates.

SAMPLING:

Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

The procedure for particulate (suspended) solids is followed and the dried residue is ignited at 600°C for one hour in a muffle furnace. The filter paper is transferred to a desiccator to cool. The ignited or ash weight is obtained as the difference between the final ignited weight and the original filter paper weight. The volume used in the ignited calculations is the volume selected for the original suspended solids calculation. Data collection, calculations and transfer of results are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 place), drying oven, muffle furnace, micro-computer system with appropriate software.

CALIBRATION: - Balance internal calibration, tare

CONTROLS AND QUALITY ASSURANCE:

Calibration: 2 S class weights
Drift: Balance zero (auto. checked every 4th measurement)
Duplicates: DUP (1 for every 10 samples)

Reporting: Maximum Significant Figures: Whole numbers
Ashed   W Value: 20   T Value: 100
Loss   W Value: 20   T Value: 100

MODIFICATIONS:

September, 1984 - Commodore computer set up for input.
September, 1988 - Direct Computer Input with Commodore computer.
June, 1989 - Microcomputer and in-house Lotus program replaced Commodore computer.

PARTICULATE SOLIDS – IGNITED

Quality Control Data from January 1 to December 31, 1993

DRIED
Analytical Range – to 6,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
18	0 – 1200	172	10.0
7	1200 – 3000	2458	28.3
16	3000 – 6000	4321	49.2

ASHED
Analytical Range – to 4,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
27	0 – 800	196	5.9
12	800 – 2000	1323	10.9
8	2000 – 4000	2441	30.9

LOSS
Analytical Range – to 4,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
19	0 – 800	145	7.8
6	800 – 2000	1555	32.9
17	2000 – 4000	2706	41.8

SOLIDS - TOTAL

IDENTIFICATION:

LIS Test Name Code:	RST	Introduced:	1980
Work Station Code:	TBSOLIDS	Units:	mg/L
Method Code:	E6035A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Sewages, surface waters, precipitation, industrial effluents and landfill leachates.

SAMPLING:

Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

A well mixed sample is evaporated in a pre-weighed dish and dried to a constant weight in an oven set at 103°C ±2°C. The increase in weight over that of the empty dish represents the total solids. Data collection, calculations and transfer of results are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 place), drying oven, micro-computer system with appropriate software.

CALIBRATION: - Balance internal calibration, tare

CONTROLS AND QUALITY ASSURANCE:

Calibration:	2 S class weights
Recovery:	LTB, REC1 (2 sets), REC2 (2 sets)
Drift:	Balance zero, daily blank, empty dishes (C1, C2)
Duplicates:	DUP (1 for every 10 samples)

Reporting:	Maximum Significant Figures: 2
	W Value: 5.0 T Value: 25.0

MODIFICATIONS:

September, 1984 - Commodore computer set up for input.
September, 1988 - Direct Computer Input with Commodore computer.
June, 1989 - In-house Lotus program replaced Commodore.

SOLIDS TOTAL

Quality Control Data from January 1 to December 31, 1993

Analytical Range − to 2000.0 mg/L

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	90	50.00007	50.00003	−0.00004	0.000032
QCB:	90	30.00000	29.99997	−0.00003	0.000026
QCA+QCB:	90	80.00007	80.00000	−0.00007	0.00005
QCA−QCB:	90	20.00007	20.00006	−0.00001	0.00003

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.000036$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
197	0 − 400	131.42	5.19298
45	400 − 1000	661.98	9.30173
15	1000 − 2000	1471.33	14.58766

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Solids-Total, QCA

QCA
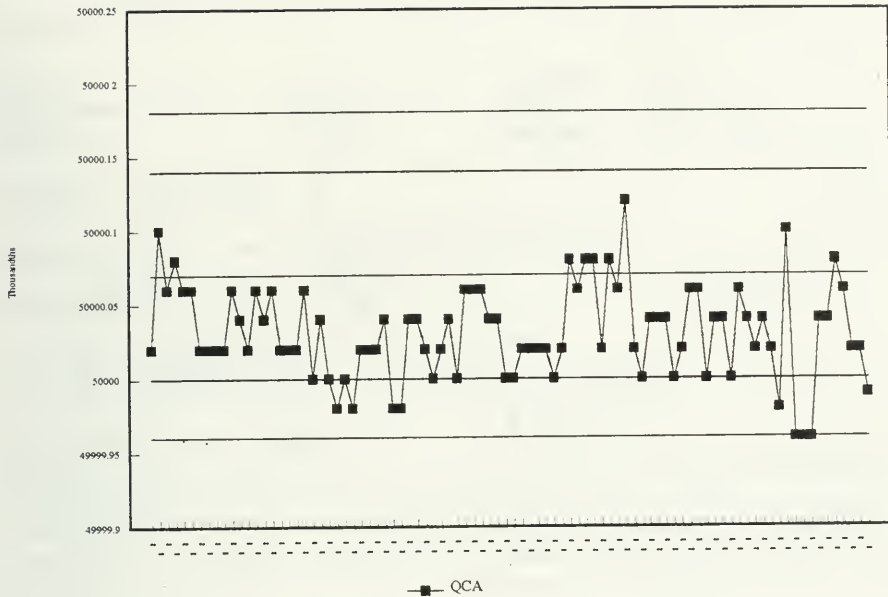
Solids-Total, QCB

QCB
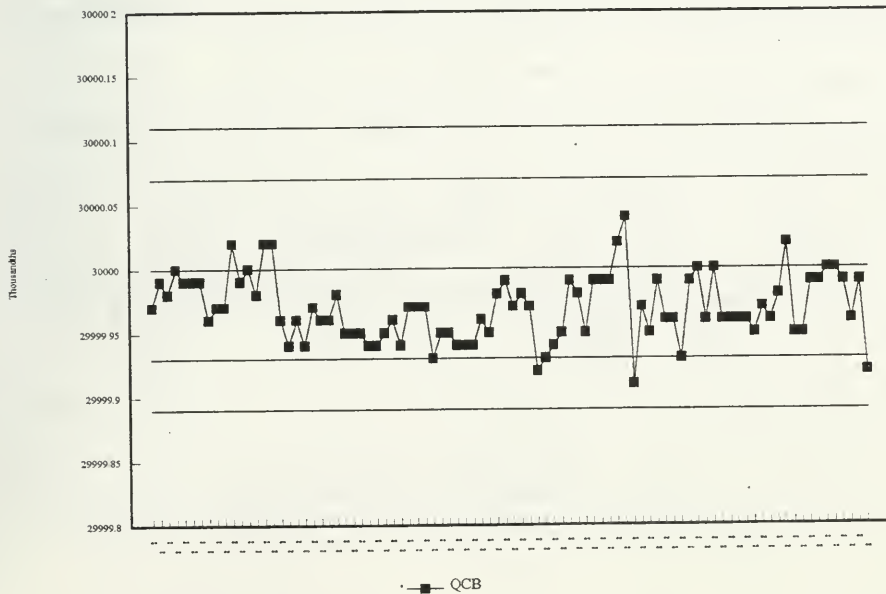
Solids-Total, QC Sum
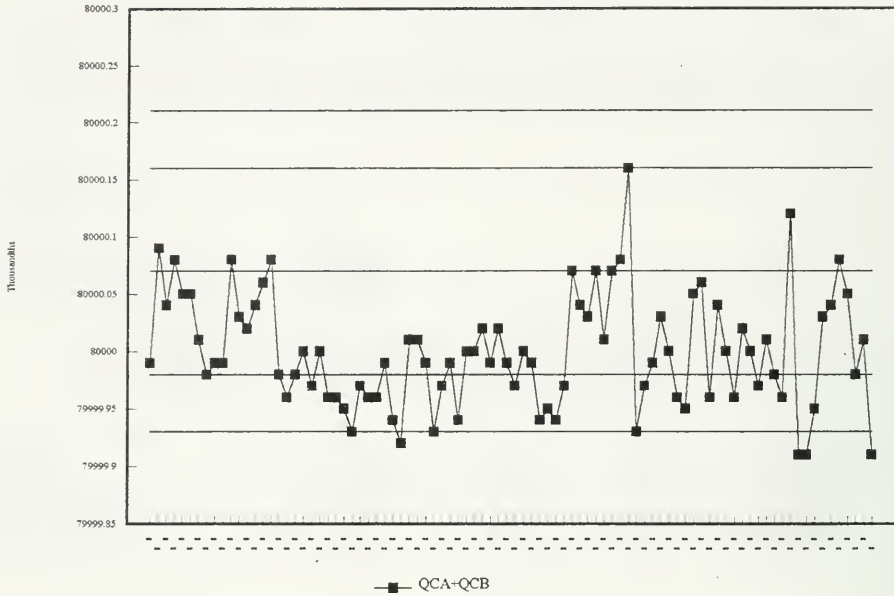
Solids-Total, QC Difference
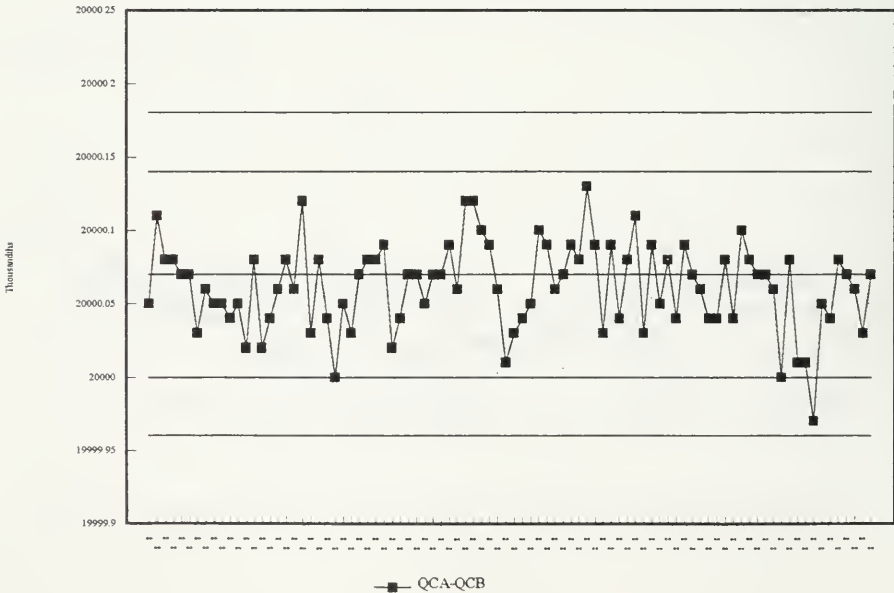
SOLIDS, TOTAL - IGNITED

IDENTIFICATION:

LIS Test Name Code:	RSTA,RSTLOI	Introduced:	1980
Work Station Code:	TBRTI	Units:	mg/L
Method Code:	E6029A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Sewages, precipitation, industrial effluents and landfill leachates.

SAMPLING:

Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:

The procedure for total solids is followed and the dried residue is ignited at 600°C for one hour in a muffle furnace. The dish is transferred to a desiccator to cool. The ignited or ash weight is obtained as the difference between the final ignited weight and the original dish weight. The volume used in the ignited calculations is the volume selected for the original total solids calculation. Data collection, calculations and transfer of results are controlled by a microcomputer system.

INSTRUMENTATION:

Balance (5 place), drying oven, muffle furnace, micro-computer system with appropriate software.

CALIBRATION: - Balance internal calibration, tare

CONTROLS AND QUALITY ASSURANCE:

Calibration:	2 S class weights
Drift:	Balance zero (auto. checked every 4th measurement)
Duplicates:	DUP (1 for every 10 samples)

Reporting: Maximum Significant Figures: Whole numbers
              Ashed   W Value: 20   T Value: 100
              Loss   W Value: 20   T Value: 100

MODIFICATIONS:

September, 1984 - Commodore computer set up for input.
September, 1988 - Direct Computer Input with Commodore computer.
June, 1989 - Microcomputer and in-house Lotus program replaces Commodore.

TOTAL SOLIDS – IGNITED

Quality Control Data from January 1 to December 31, 1993

DRIED
Analytical Range – to 20,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
2	0 – 4000	1939	33.0
13	4000 – 10000	6709	131.0
15	10000 – 20000	11981	122.1

ASHED
Analytical Range – to 10,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
5	0 – 2000	1373	10.1
13	2000 – 5000	3380	30.2
11	5000 – 10000	5532	105.1

LOSS
Analytical Range – to 20,000.0 mg/L

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
9	0 – 4000	2777	63.8
21	4000 – 10000	6974	49.3
2	10000 – 20000	13028	482.2

SULPHATE

IDENTIFICATION:

LIS Test Name Code:	SSO4UR	Introduced:	1978
Work Station Code:	TBSSO4UR	Units:	mg/L as SO_4
Method Code:	E6032B	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, precipitation, snow, industrial effluent and landfill leachate.

SAMPLING:

Container: PET or glass
Preservative: Refrigerate at 4°C

ANALYTICAL PROCEDURE:
Using a sodium bicarbonate/sodium carbonate eluent, sulphate is separated with automated ion suppression chromatography utilizing a conductivity detector, for a response vs. time chromatogram.

INSTRUMENTATION:
Dionex 2000i/SP Suppressed Ion Chromatography Module with auto-sampler, computer and controlling software.

CALIBRATION: - Quadratic
                   - 6 Standards, 2-50 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration: LTB, QCA, QCB, QCC
Drift: SENS.CHK (2 every 20 samples)
Duplicates: 3 DUPS (analyzed at beginning of run)

Reporting: Maximum Significant Figures: 2
            W Value: 0.5   T Value: 2.5

CAEAL Accredited, LRTAP and QM Blind Audit participants

MODIFICATIONS:

1978-1984 -Sulphate analyzed using $BaCl_2$-methylthymol blue colourimetric method.
1984-1990 -Sulphate analyzed using Wescan Ion Chromatography module.
Aug. 1990 -Chromatography Module changed to Dionex 2000i/SP.
May 1991 -Dionex Autosampler was added to replace Technicon Sampler IV.

SULPHATE

Analytical Range – to 50.0 mg/L as SO4

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	93	45	45.197	0.197	0.2811
QCB:	93	15	14.983	−0.017	0.0222
QCC:	93	5	5.018	0.018	0.1048
QCA+QCB:	93	60	60.180	0.180	0.3687
QCA−QCB:	93	30	30.214	0.214	0.3477
QCB+QCC:	93	20	20.001	0.001	0.2859
QCB−QCC:	93	10	9.965	−0.035	0.1977

For 1993 Control Charts:

$$Sw\ (A-B) = \quad 0.7148$$
$$Sw\ (B-C) = \quad 0.2639$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
117	0 − 10	4.405	0.0998
68	10 − 25	15.217	0.2511
26	25 − 50	35.368	0.5561

Sulphate, QCA

— QCA

Sulphate, QCB

— QCB

151
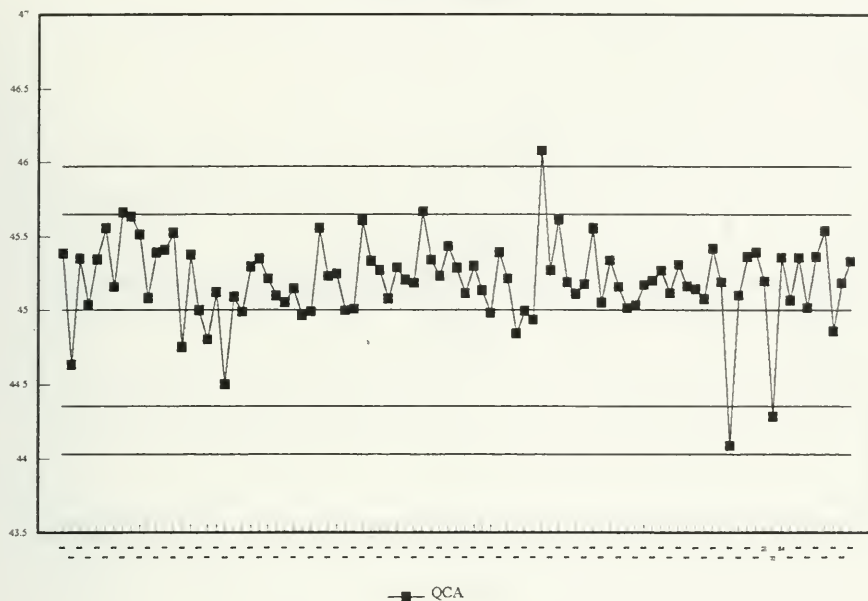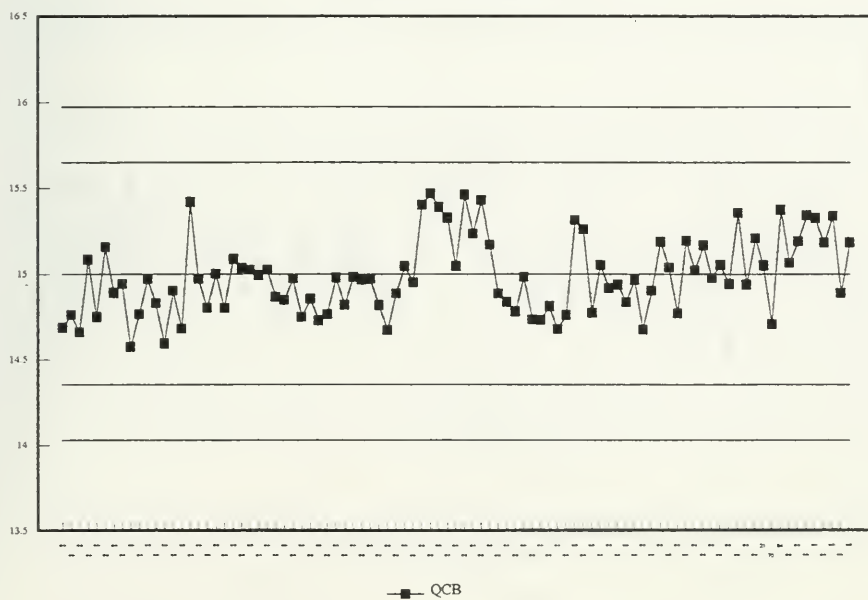
Sulphate, QCC

QCC

Sulphate, QC Sum

QCA+QCB
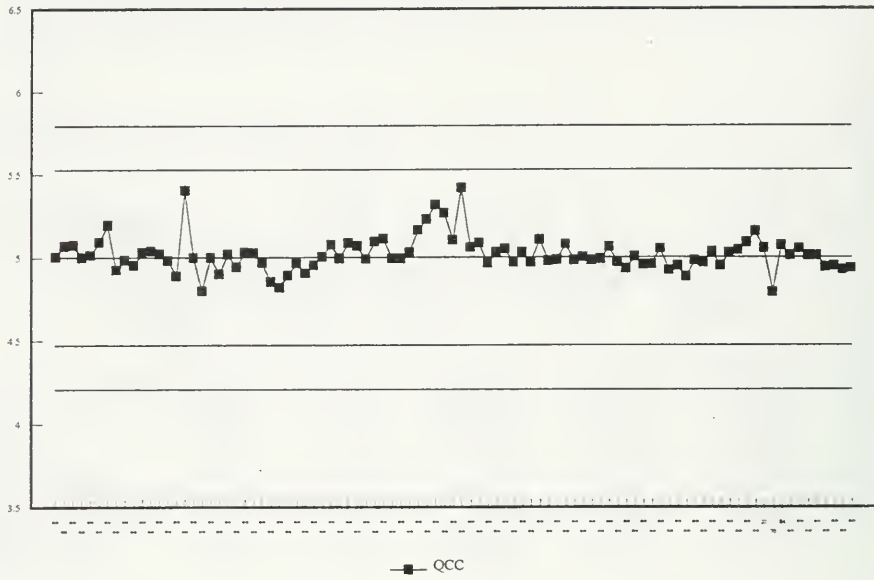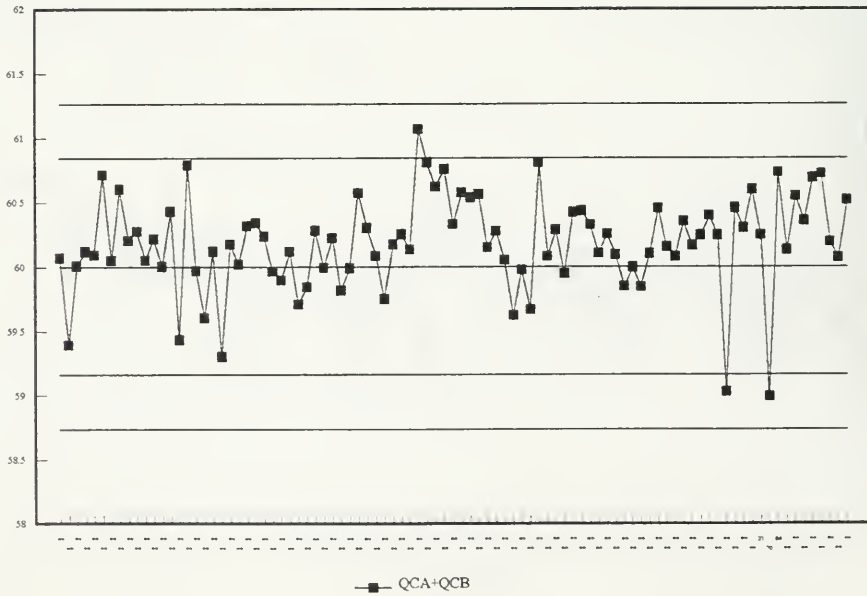
Sulphate, QC Difference

QCA-QCB

Sulphate, QCC Sum

QCB+QCC
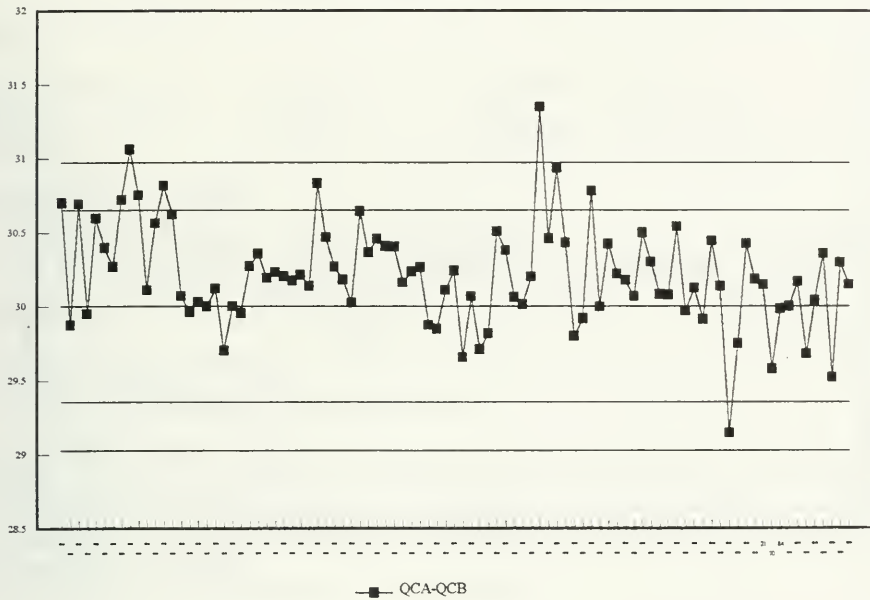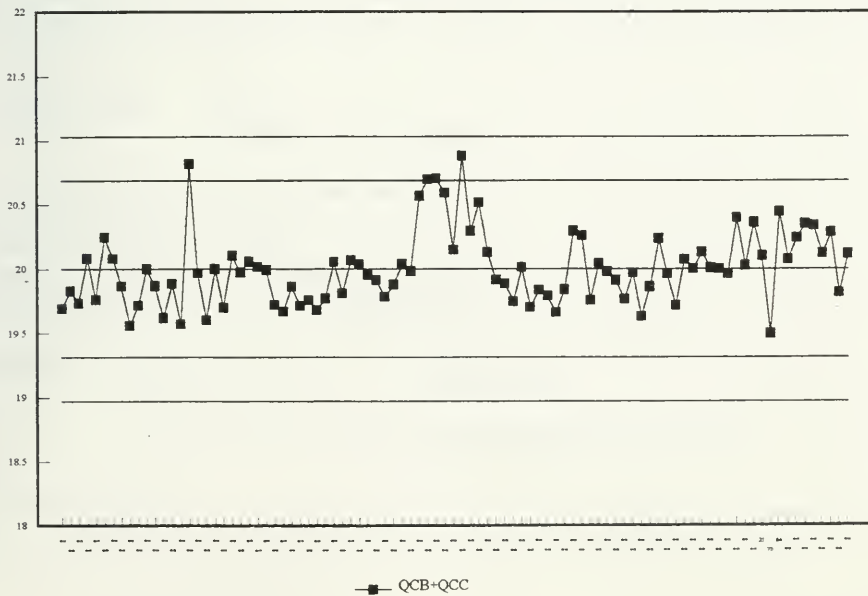
Sulphate, QCC Difference

QCB-QCC
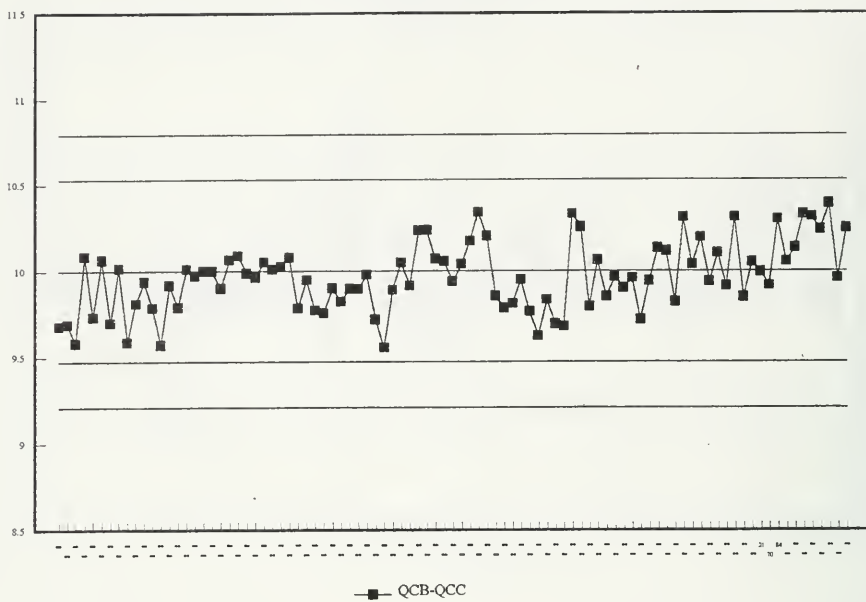
TOTAL KJELDAHL NITROGEN

IDENTIFICATION:

LIS Test Name Code:	NNTKUR	Introduced:	1978
Work Station Code:	TBTNTP	Units:	mg/L as N
Method Code:	E6026A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface and domestic waters, precipitation, sewages, landfill leachates and industrial effluents.

SAMPLING:

Container: Glass or PET jar.
Preservative: Refrigerate at 4°C.

ANALYTICAL PROCEDURE:
Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters set at 200°C and 360°C. The digested sample is then neutralized and analyzed for ammonia species using phenate-hypochlorite colourimetry which determines both organic and ammonia forms of nitrogen.
N.B. Total Phosphorus is determined simultaneously.

INSTRUMENTATION:
Automated continuous flow system, Technicon AAII with a 37°C heating bath. Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Data capture and processing via a multi-stage microcomputer system.

CALIBRATION: - Linear
                     - 7 Standards   0 - 1.60 mg/L

CONTROLS AND QUALITY ASSURANCE:

Calibration:	LTB, QCA, QCB,
Drift:	BLK every 10 samples, CHK (100%) every 20 samples
Duplicates:	DUP (3 per run, run at beginning)

Reporting: Maximum Significant Figures: 2
               W Value: 0.01   T Value: 0.05

               CAEAL Accredited, LRTAP and QM Blind Audit participant

MODIFICATIONS:

February 1989 - Both channels went to microcomputer control with DCI software.

TOTAL KJELDAHL NITROGEN

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 1.60 mg/L as N

Calibration Control:

|	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	88	1.5	1.510	0.010	0.0113
QCB:	88	0.5	0.506	0.006	0.0093
QCA+QCB:	88	2.0	2.016	0.016	0.0166
QCA−QCB:	88	1.0	1.004	0.004	0.0124

For 1993 Control Charts:

Sw (A−B) = 0.0125

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
123	0.00 − 0.32	0.160	0.0183
71	0.32 − 0.80	0.508	0.0198
28	0.80 − 1.60	1.083	0.0243

Total Kjeldahl Nitrogen, QCA

QCA
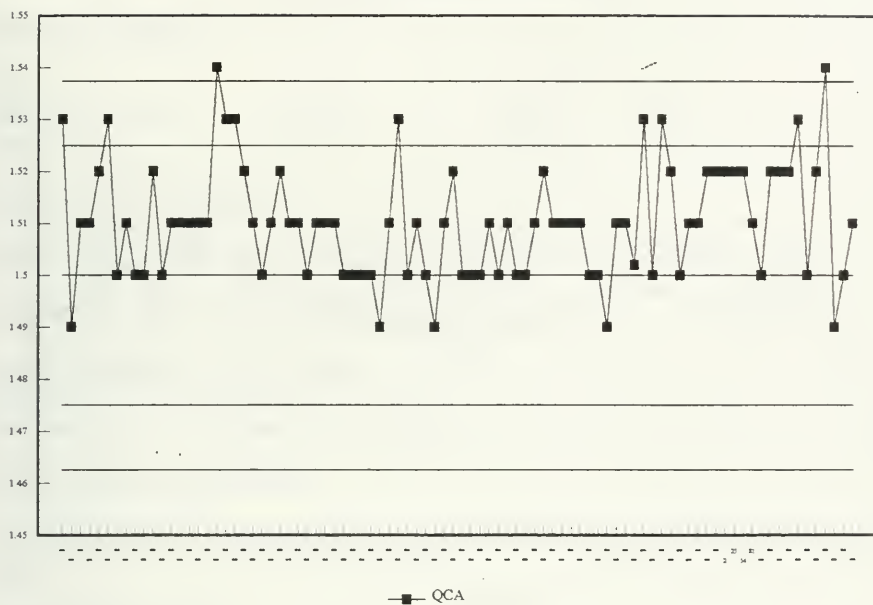
Total Kjeldahl Nitrogen, QCB

QCB

157
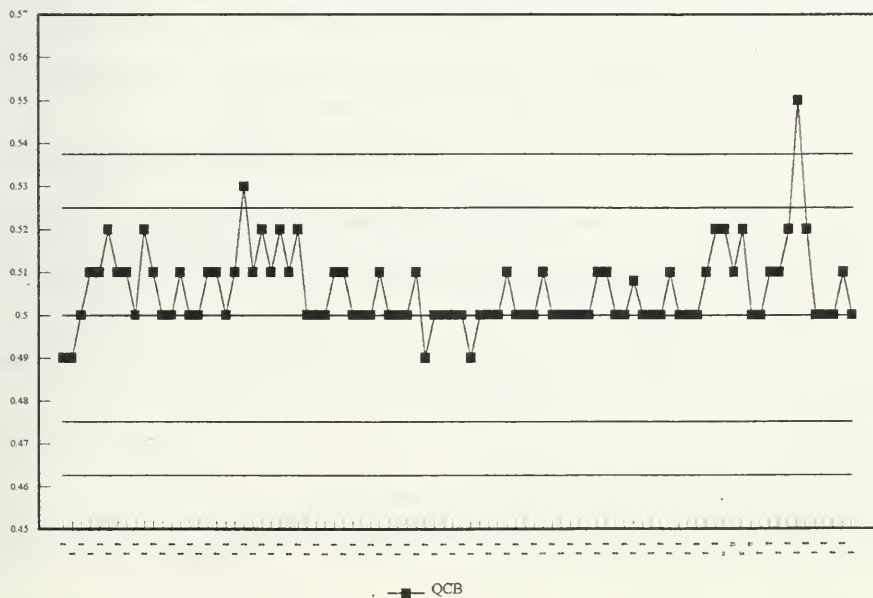
Total Kjeldahl Nitrogen, QC Sum

QCA+QCB
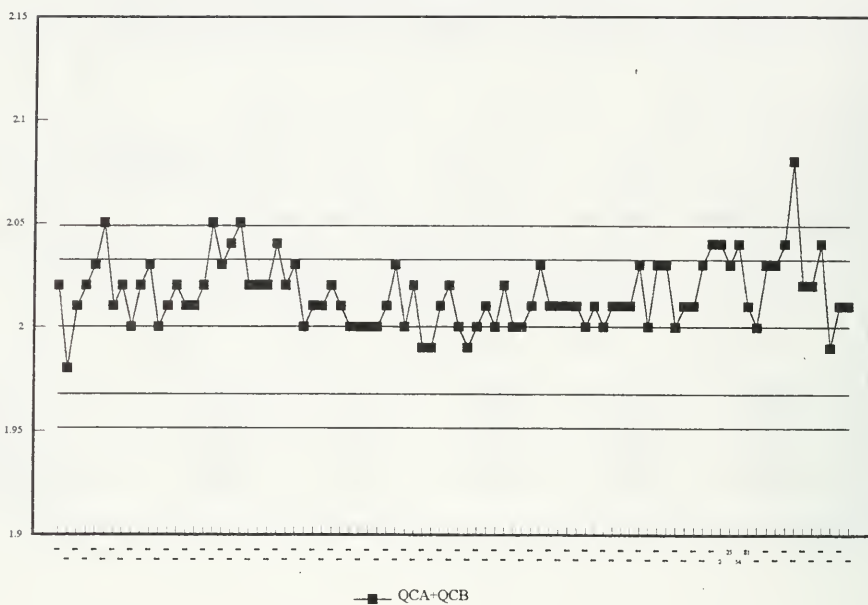
Total Kjeldahl Nitrogen, QC Difference

QCA-QCB
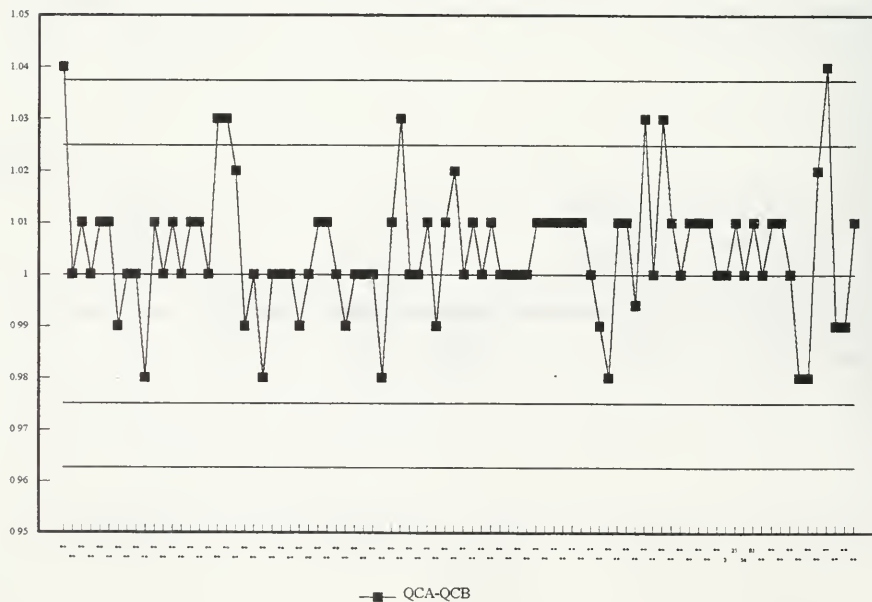
TURBIDITY

IDENTIFICATION:

LIS Test Name Code:	TURB	Introduced:	1978
Work Station Code:	TBTURB	Units:	FTU
Method Code:	E6011A	Section:	Water Quality

SAMPLE TYPE/MATRIX:

Surface water, domestic water, precipitation, industrial effluent and landfill leachate.

SAMPLING:

Special Instructions:	Avoid freezing
Container:	PET or glass
Preservative:	Refrigerate at 4°C in dark

ANALYTICAL PROCEDURE:

Samples are placed in the turbidimeter and results in FTU are read directly from the digital output.

INSTRUMENTATION:

Hach Ratio Turbidimeter, Model 18900. Turbidity measurements are based on light scattering at 90 degrees plus or minus 30 degrees of rotation.

CALIBRATION: -Calibrated every 6 months using 11 Stock Formazin
-Standards ranging from 0.4 to 180 FTU.

CONTROLS AND QUALITY ASSURANCE:

Calibration:	QCA, QCB (Sealed Hach 'Gelex' Standard)
Drift:	QCA, QCB - at end of run
Duplicates:	DUPS (1 for every 15 samples, run at beginning)

Reporting:	Maximum Significant Figures: 2
	W Value: .02 T Value: .10

MODIFICATIONS:

March 1987 - New Hach Ratio Turbidimeter put into use to replace older Hach model.
January 1990 - New Hach 'Gelex' Standards were put into use after re-calibration of the Hach Model 18900 Turbidimeter.

Quality Control Data from January 1 to December 31, 1993

Analytical Range — to 10.0 FTU

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCA:	73	15.777	15.756	−0.021	0.0687
QCB:	73	1.547	1.544	−0.003	0.0236
QCA+QCB:	73	17.324	17.300	−0.024	0.0823
QCA−QCB:	73	14.230	14.212	−0.018	0.0615

For 1993 Control Charts:

$$Sw (A-B) = \quad 0.1009$$

Duplicates:

Number of Data Pairs	Sample Conc Span	Mean Value	Standard Deviation
138	0 − 2	0.783	0.0642
37	2 − 5	3.210	0.2102
28	5 − 10	6.850	0.3048

Turbidity, QCA

Turbidity, QCB

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Turbidity, QC Sum

- ■— QCA+QCB
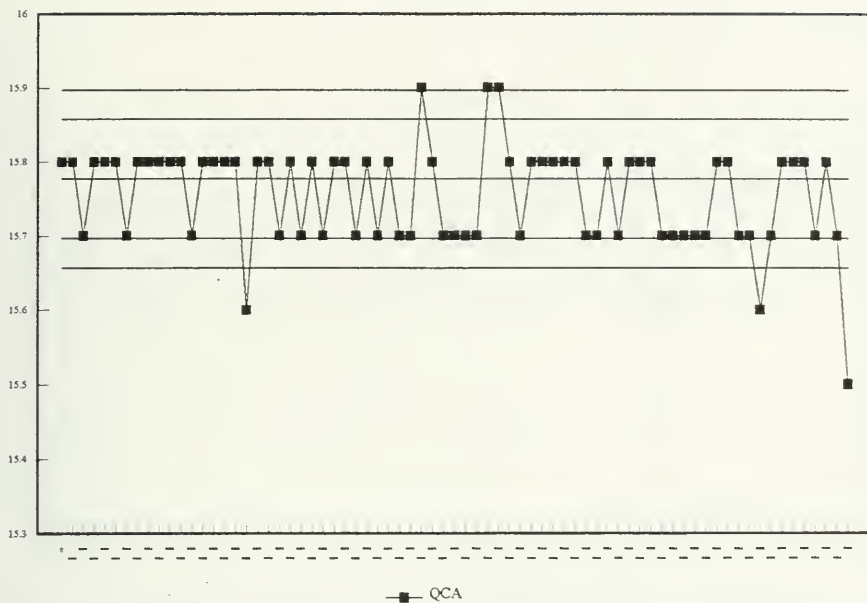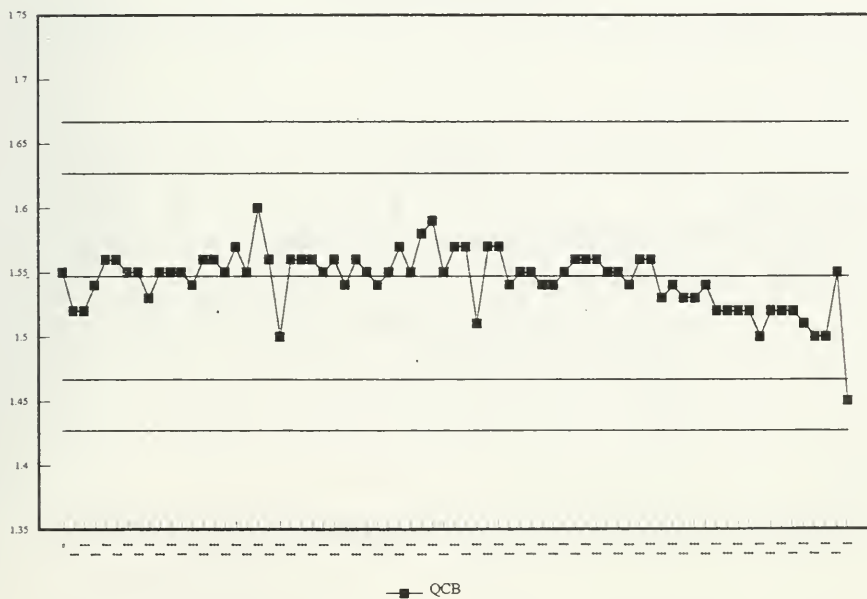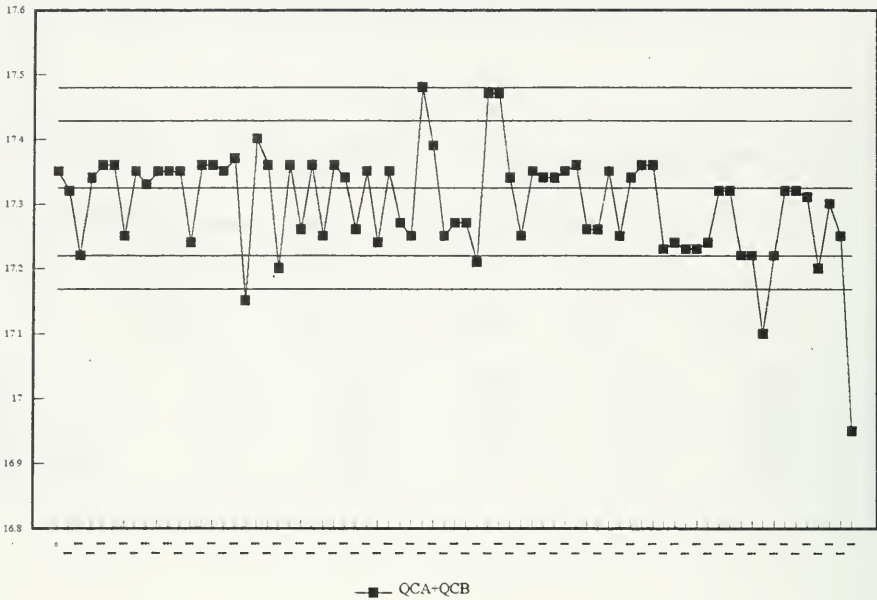
Turbidity, QC Difference

- ■— QCA-QCB

162
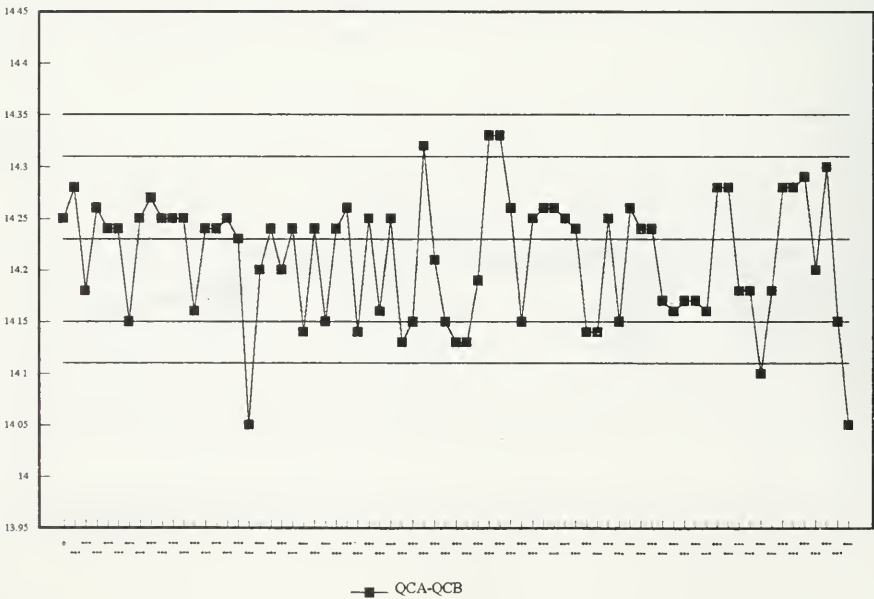
5.0   Trace Contaminants Performance Summaries

BORON

IDENTIFICATION:

Method Title: The Determination of Boron in Water by ICP-AES.

Work Station Code:	TBBORON	Method Introduced:	June, 1988
Method Code:	E6036A	Current Revision:	July 1993
		Section:	Trace Contaminants

PARAMETER:

Element	LIS Code	W (ug/L)	T (ug/L)
Boron	BBUT	5	25

SAMPLE TYPE/MATRIX:

Domestic water, surface water, groundwater, industrial wastes, landfill, test wells, etc.

ANALYTICAL PROCEDURE:

Sample is aspirated without pretreatment into the analytical instrument. Concentration is determined by intensity of light emission at a specific boron wavelength..

INSTRUMENTATION:

Thermo Jarrell Ash ICAP61 Inductively Coupled Plasma Spectrometer; Linear 2-point calibration, 0-2.5 mg Boron/L.

QUALITY ASSURANCE:

Controls:	Blank, ICAP7 (1.0 ug/mL)
Ref. Material:	ICAP7 (EPA)
Drift:	QCBOR (0.5 ug/mL) analyzed every 10 samples
Duplicates:	1 per 10 samples
Interlabs:	MOEE Blind Audit Program Great Lakes Action Program (GLAP, 2x annually)
Reporting:	Units: ug/L Sig. Figures: 2

BORON (TOTAL) — BBUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range — 5–50,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% of Target	% Rel. Std. Dev.
QCBOR:	120	0.5	0.497	99.4	2.10

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
70	5	500	50.	3.0
5	500	5000	890.	7.1
N/A	5000	50000	—	—

Detection Limit (DL) = 5 ug/L

Boron

Time

QCBOR ___ Ave.

Boron

Time

ICAP−7 ___ Ave.

166
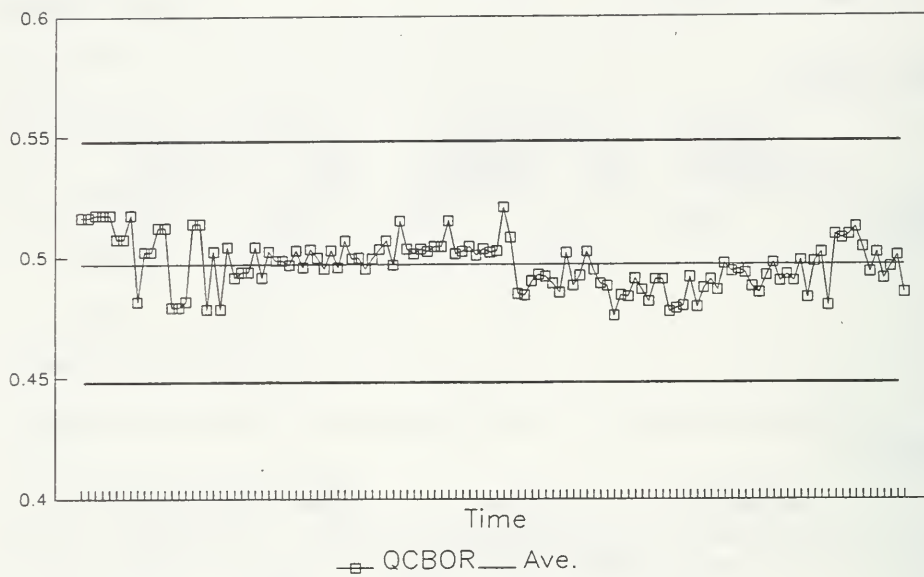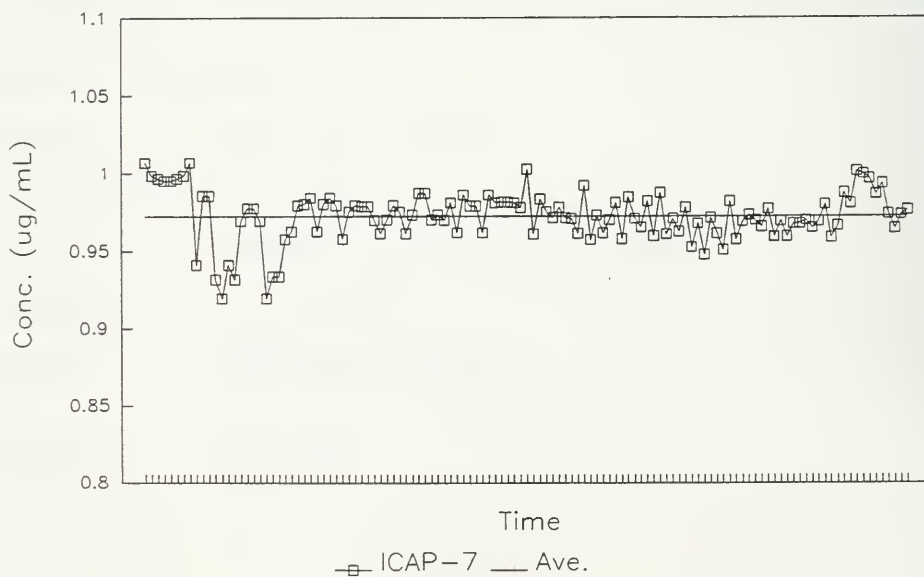
HYDRIDES: ANTIMONY, ARSENIC, AND SELENIUM

IDENTIFICATION:

Method Title: The Determination of Arsenic, Selenium and Antimony in Surface and Drinking Water by Hydride AAS.

Work Station Code:	TBHYDW	Date Introduced:	1980
Method Code:	E6038A	Current Revision:	Nov. 1992
		Section:	Trace Contaminants

PARAMETER:

Element	LIS Code	W (ug/L)	T (ug/L)
Arsenic	ASUT	.2	1.0
Selenium	SEUT	.2	1.0
Antimony	SBUT	.2	1.0

SAMPLE TYPE/MATRIX:
Surface, drinking waters, landfill leachates, and industrial effluent.

ANALYTICAL PROCEDURE:
Samples are digested in oxidizing acid mixtures to oxidize all forms. It is reduced by sodium borohydride and swept into a heated quartz tube by argon carrier gas. Concentration is determined by flameless atomic absorption spectrometry.

INSTRUMENTATION:

Varian AA-6 Automated AAS with quartz cell; Logarithmic, 6-Standard calibration, 0.0-40.0 ug/L

CONTROLS AND QUALITY ASSURANCE:

Controls:	Blank, QCA, QCB
Ref. Material:	ERA PP/CLP Trace Metals Standard
Drift:	10.0 and 30.0 ug/L standards every 10 samples
Duplicates:	1 for every 13 samples, throughout run
Interlabs:	MOEE Blind Audit Program. Great Lakes Action Program (2x annually)
Reporting:	Units: ug/L (ppb) Sig. Figures: 2

ANTIMONY (TOTAL) – SBUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.2 – 40 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCA:	24	40.000	40.465	101.2	2.0
QCB:	24	10.000	10.934	109.3	4.5
QCA+QCB:	24	50.000	51.399	102.8	1.5
QCA−QCB:	24	30.000	29.530	98.4	3.6

For 1993 Control Limits:

$$Sw (A-B) = \quad 1.383$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
95	0.2	8.0	0.40	0.1347
−	8.0	20.0	−	−
0	20.0	40.0	−	−

Detection Limit (DL) = | 0.2 ug/L |

Antimony
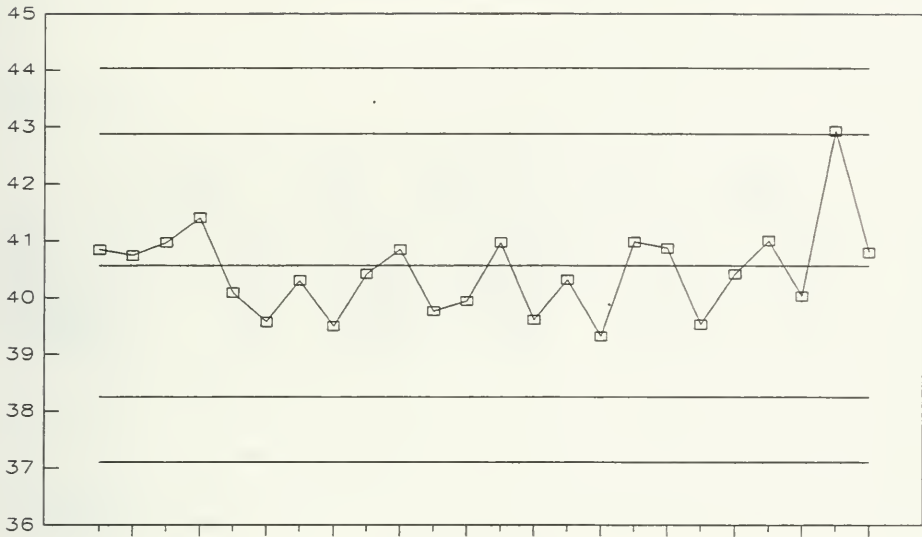
QCA

Antimony
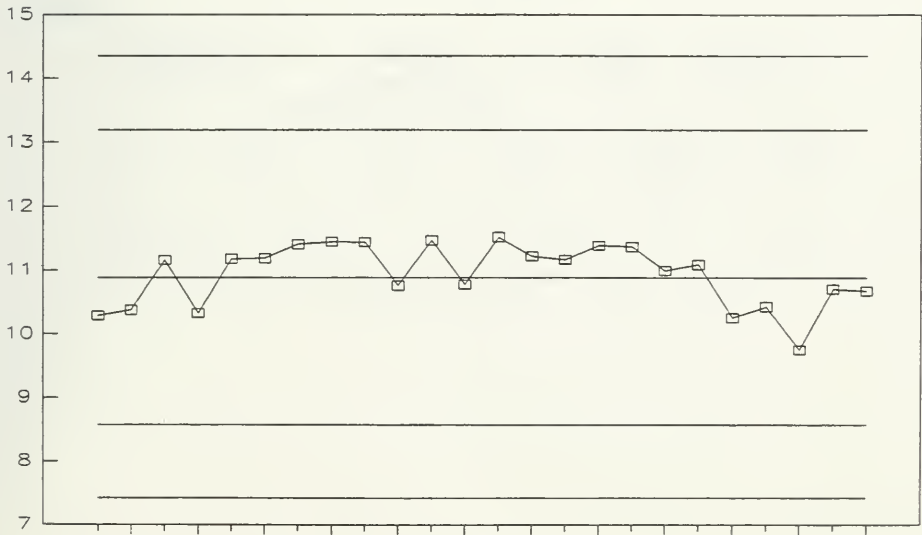
QCB

Antimony, QC Difference

QCA - QCB

Antimony, QC Sum
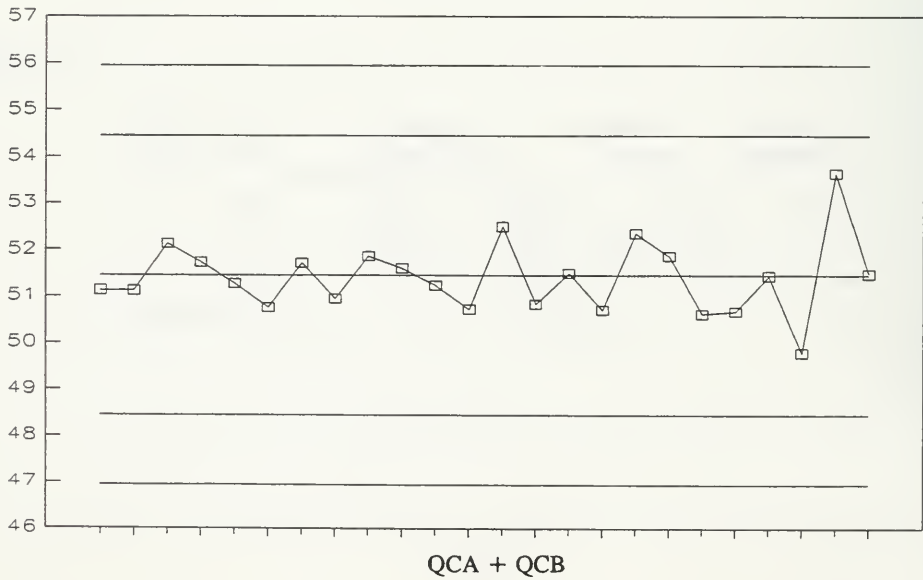
QCA + QCB

ARSENIC (TOTAL) — ASUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.2 — 40 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCA:	53	40.000	39.878	99.7	1.7
QCB:	53	10.000	10.762	107.6	4.4
QCA+QCB:	53	50.000	50.641	101.3	1.9
QCA−QCB:	53	30.000	29.116	97.1	2.4

For 1993 Control Limits:

Sw (A−B) =   0.838

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
146	0.2	8	0.85	0.26
8	8	20	12.1	0.91
2	20	40	28.9	1.71

Detection Limit (DL) =   0.2 ug/L

Arsenic
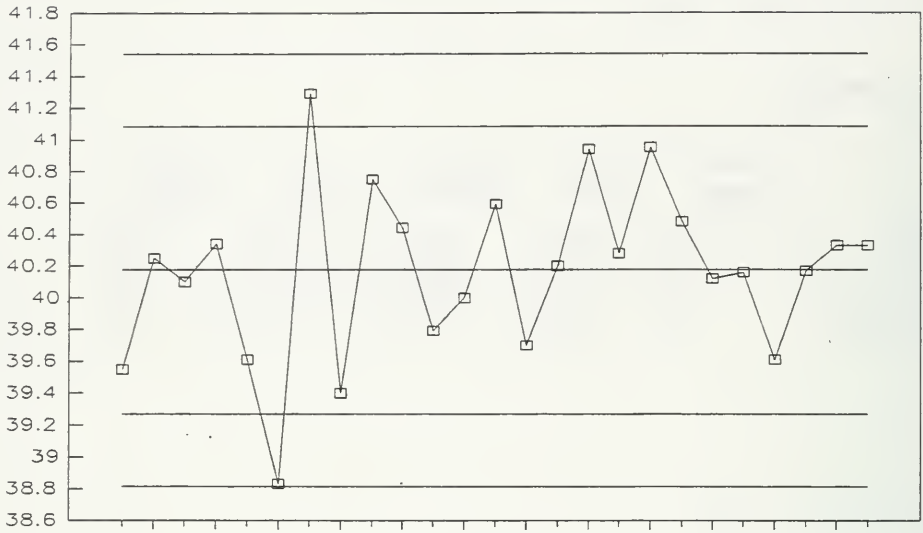
QCA

Arsenic
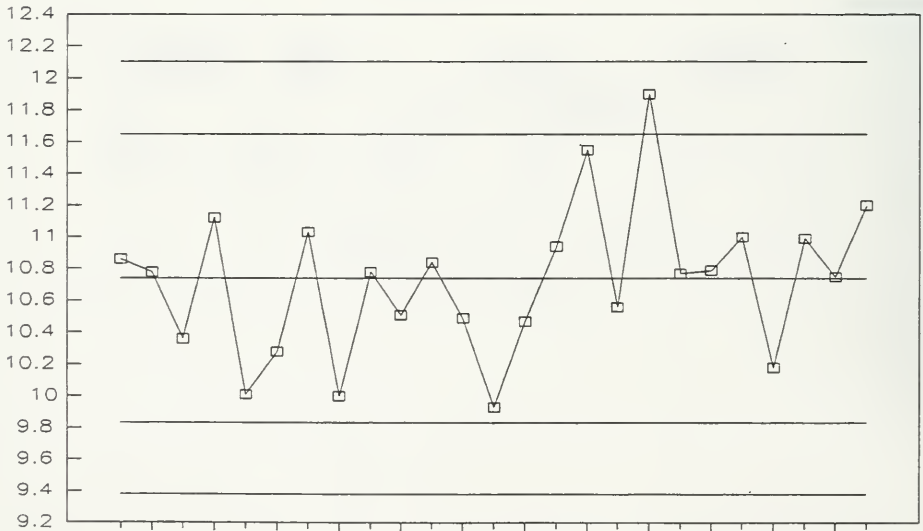
QCB

Arsenic, QC Difference
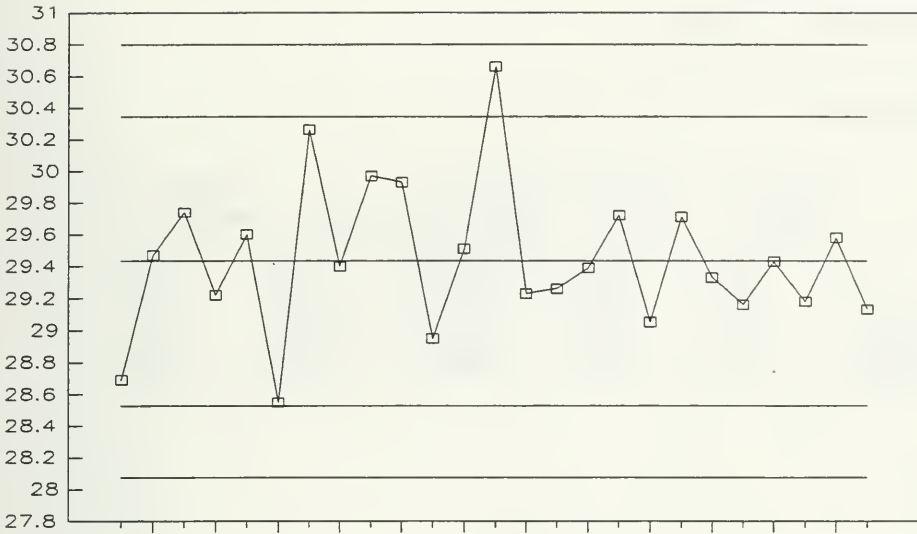
QCA - QCB

Arsenic, QC Sum

QCA + QCB

173
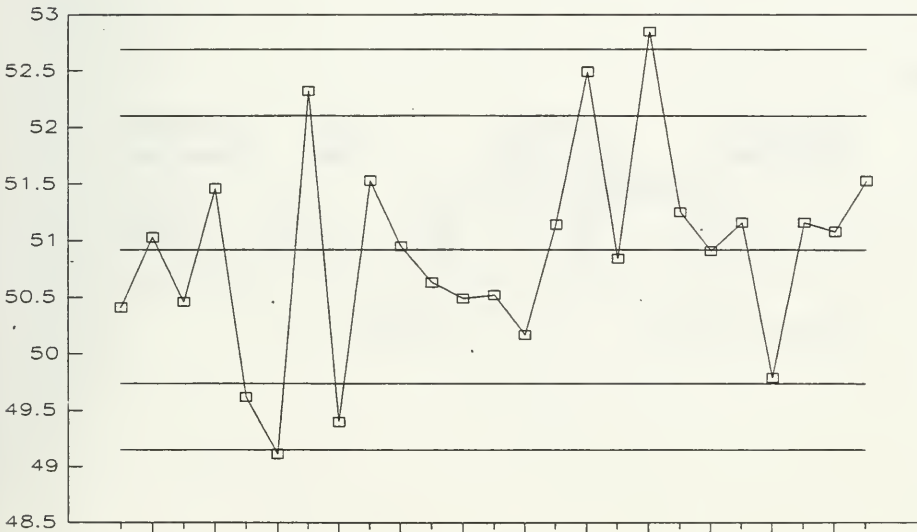
SELENIUM (TOTAL) — SEUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.2 – 40 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCA:	31	40.000	39.831	99.6	1.6
QCB:	31	10.000	10.486	104.9	3.8
QCA+QCB:	31	50.000	50.317	100.6	1.5
QCA−QCB:	31	30.000	29.345	97.8	2.5

For 1993 Control Limits:

$$Sw (A-B) = 1.6256$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
59	0.2	8	0.85	0.1738
3	8	20	11.4	0.4
0	20	40	–	–

Detection Limit (DL) = 0.2 ug/L

Selenium
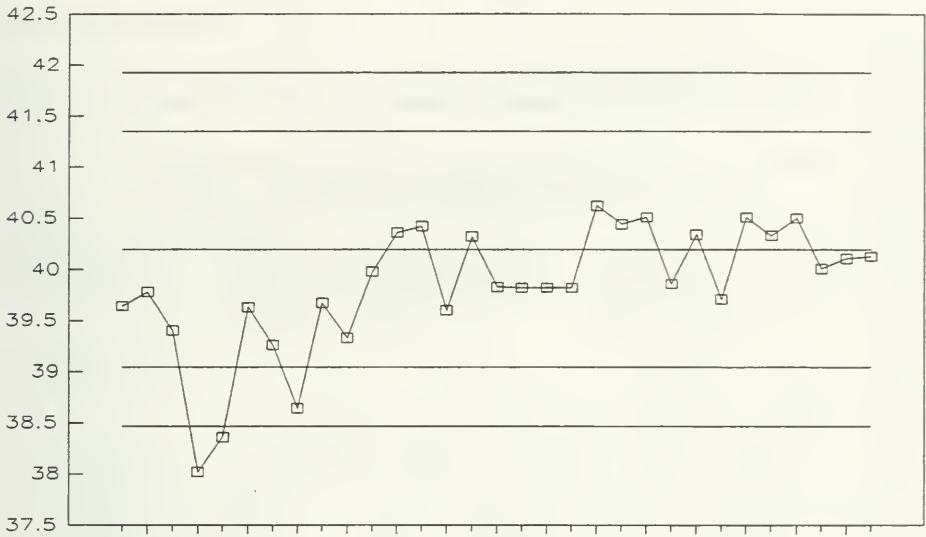
QCA

Selenium
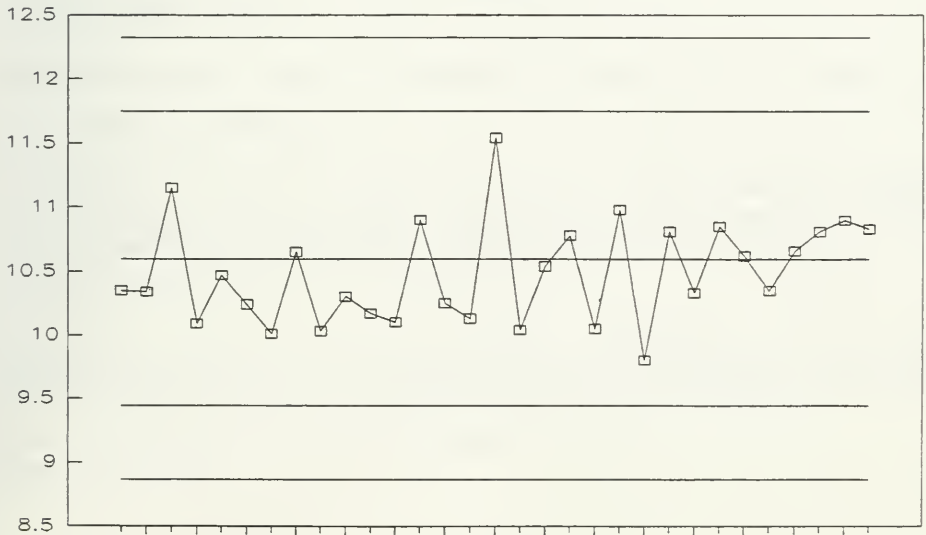
QCB

Selenium, QC Difference

QCA - QCB

Selenium, QC Sum

QCA + QCB
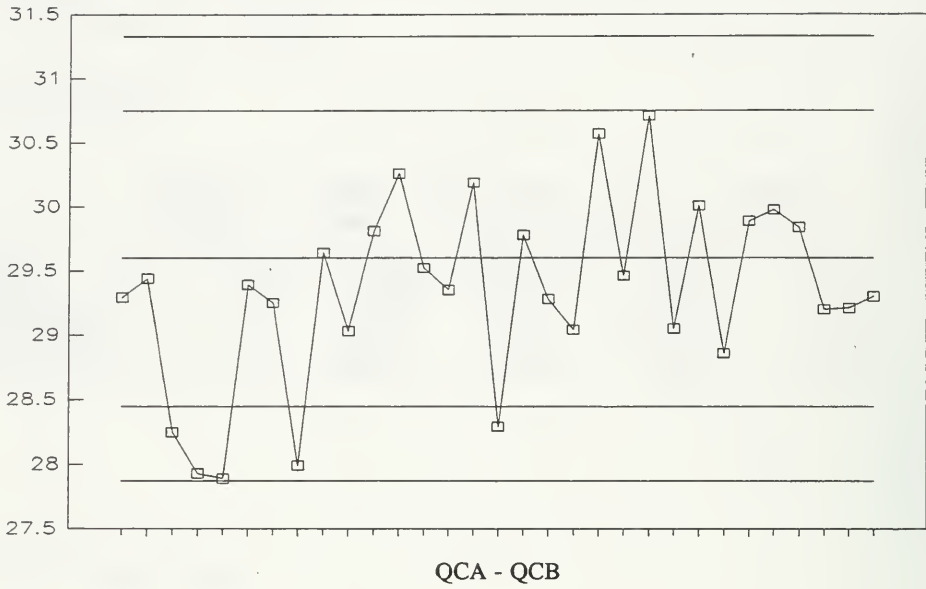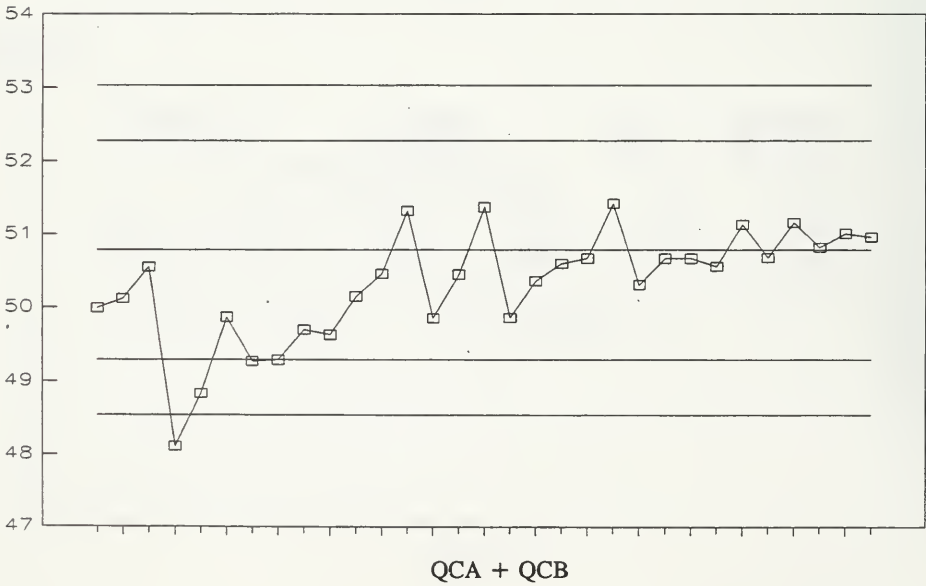
MAJOR CATIONS

IDENTIFICATION:

Method Title: The Determination of Sodium, Potassium, Calcium, and Magnesium in Water, Sewage, Leachates and Industrial Wastes by Sequential ICP-AES.

The Determination of Sodium, Potassium, Calcium and Magnesium in Water, Sewage, Leachates and Industrial Wastes by Flame-AAS.

Work Station Code: TBSOFT   Date Introduced: July 1980 (AA); November, 1991 (ICP)
Method Code: E6047A (ICP)   Current Revision: July 1993
   E6039A (AA)   Section: Trace Contaminants

PARAMETERS:

Element	LIS Code	W (mg/L)	T (mg/L)
Sodium	NAUR	.05	.25
Potassium(ICP)	KKUR	0.5	2.5
Potassium (AA)	KKUR	0.02	.10
Calcium	CAUR	.05	.25
Magnesium	MGUR	.02	.10

SAMPLE TYPE/MATRIX:

Sewage, Surface Waters, Landfills, Drinking Waters, Leachates, and Industrial Waters.

ANALYTICAL PROCEDURE:

Concentrations in solution are determined by inductively coupled argon plasma emission spectroscopy (direct aspiration). Samples containing less than 1 ppm potassium are analyzed by atomic absorption spectroscopy, which has a lower detection limit for potassium.

INSTRUMENTATION:

Atomic Emission Spectrometer, Thermo Jarrell Ash Atomscan 25 with Thermo Jarrell Ash Model TJA 300 Autosampler; Linear, 2 point calibration with automatic line switching on Mg and Ca.

Varian Model 1475 Atomic Absorption Spectrometer with Varian Model 55 Autosampler.

CONTROLS AND QUALITY ASSURANCE:

Control: Blank, Veg 7, QCB

Ref.Material: SLRS-2 (National Research Council, Canada)
                    ERA PP/CLP Cations Standard

Drift: Veg 7 run every 10 samples

Duplicates: 1 every 20 samples

Interlabs: QM Office Blind Audit Program-bimonthly
                    LRTAP (Fisheries and Oceans, Canada, 3x annually)
                    Great Lakes Action Program (GLAP, 2x annually)
                    CAEAL Certification Round Robin (2x annually)

Reporting: Units: ppm (mg/L)
                    Sig. Figures: 2

REMARKS: See Section 1.2 for parameters that are accredited with CAEAL.

CALCIUM (REACTIVE) — CAUR

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.05 – 500 mg/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% of Target	% Rel. Std. Dev.
VEG7:	242	40.0	40.180	100.5	2.1
QCB:	44	1.000	1.040	104.0	2.2

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
36	0.05	5	2.22	0.066
88	5	50	19.	0.34
59	50	500	125	3.7

Detection Limit (DL) =   0.05 mg/L

Calcium
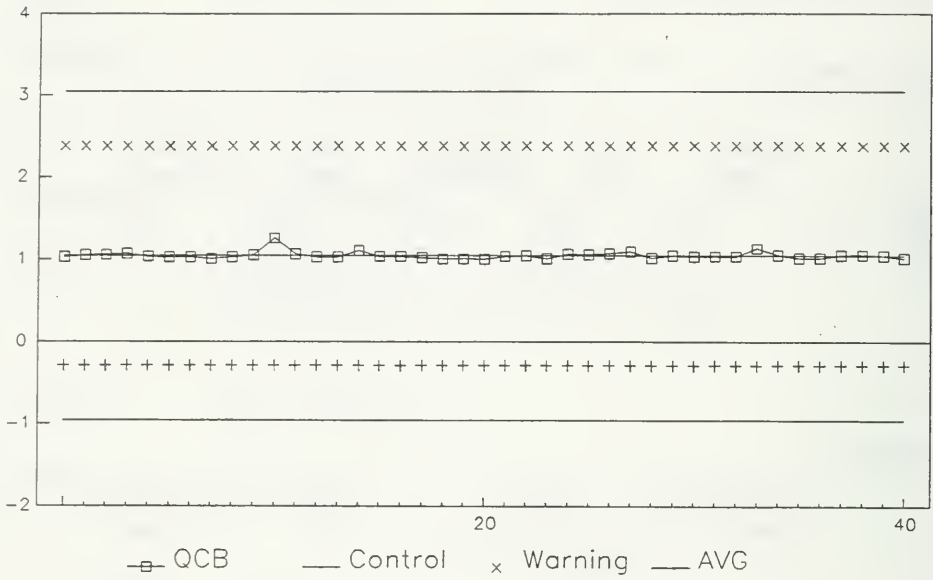
Calcium
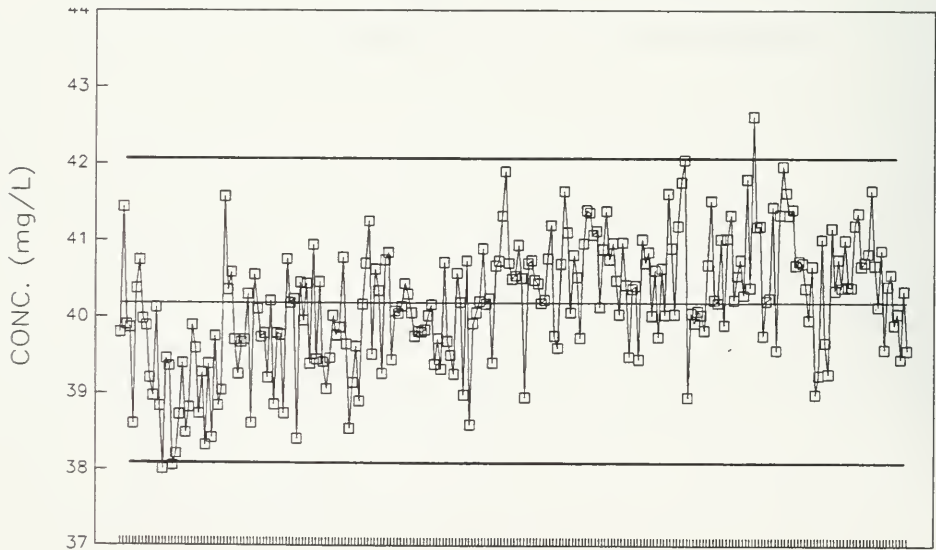
VEG7

MAGNESIUM (REACTIVE) – MGUR

Analytical Range: 0.05 – 200 mg/L

Calibration Control:

	Number of Data	Target Conc.	Avg. Conc. Measured	% of Target	% Rel. Std. Dev.
VEG7:	249	10.0	10.050	100.5	2.7
QCB:	40	0.100	0.102	102.0	2.1

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
62	0.05	5	0.93	0.058
76	5	50	6.7	0.16
47	50	200	61.	1.6

Detection Limit (DL) =   0.05 mg/L

Magnesium
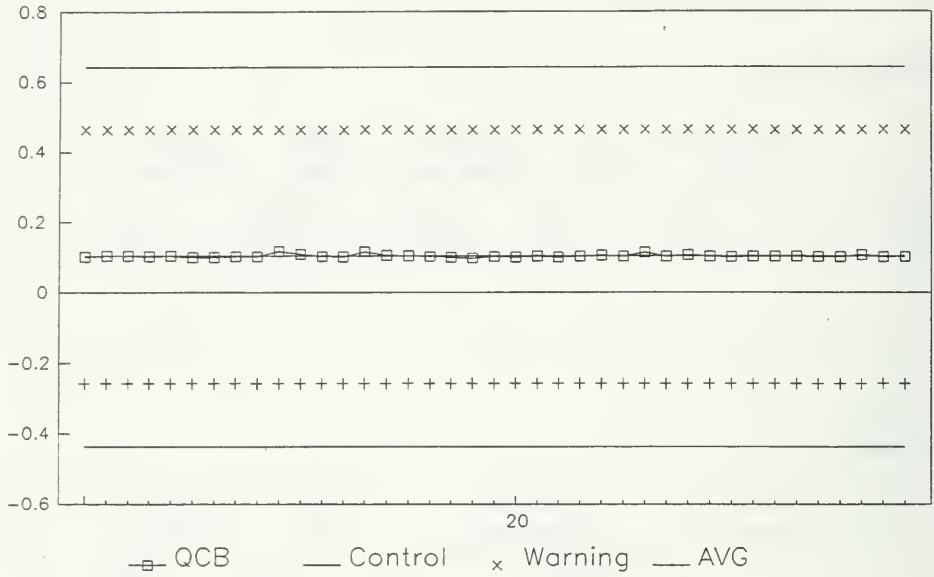
Magnesium
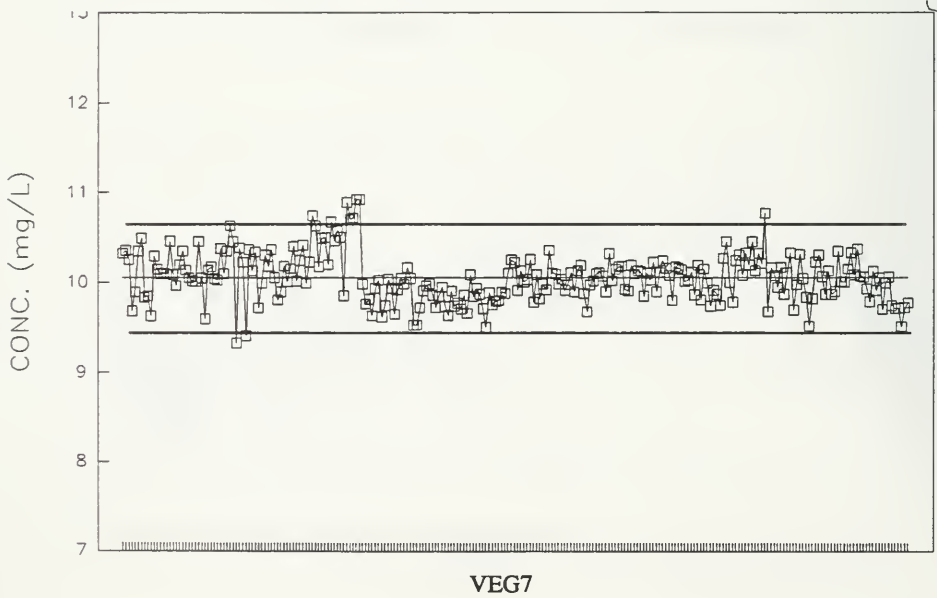
VEG7

POTASSIUM (REACTIVE) — KKUR

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.05 − 250 mg/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% of Target	% Rel. Std. Dev.
VEG7:	220	20.0	19.190	96.0	2.4
QCB:	46	2.000	2.060	103.0	5.8

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
181	0.05	5	2.7	0.17
10	5	50	86.	2.15
N/A	50	250	—	—

Detection Limit (DL) =   0.05 mg/L

Potassium
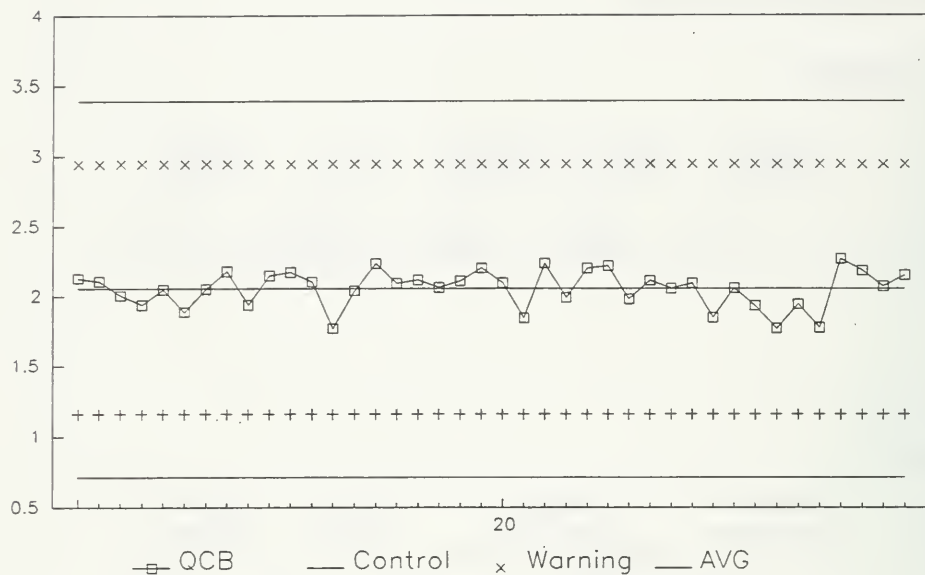
Potassium
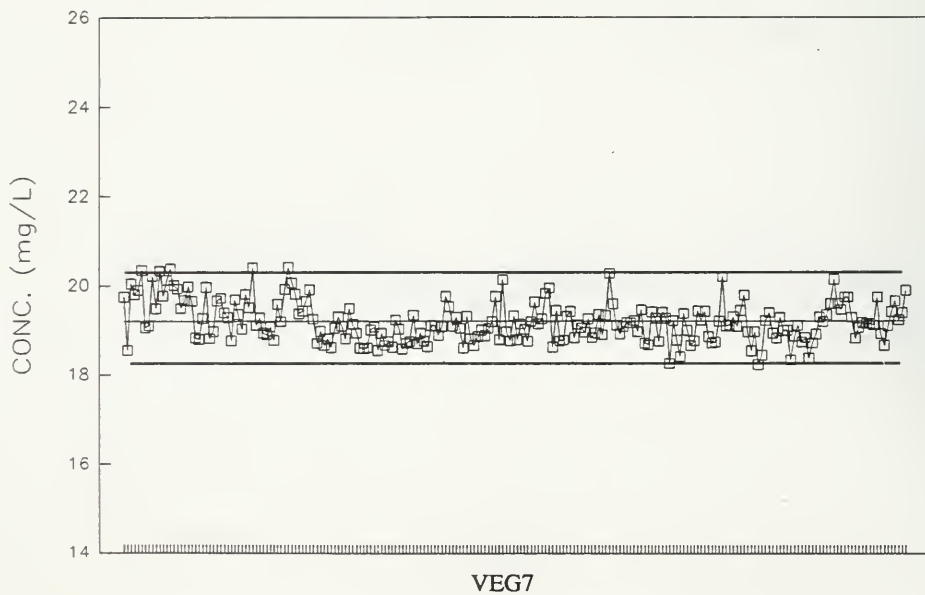
VEG7

SODIUM (REACTIVE) — NAUR

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.05 — 500 mg/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% of Target	% Rel. Std. Dev.
VEG7:	321	20.0	19.9	99.6	2.6
QCB:	50	0.5	0.5	106.0	4.8

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
133	0.05	5	2.32	0.09
113	5	50	19.	0.39
49	50	500	216.	4.7

Detection Limit (DL) = 0.05 mg/L

Sodium


```
        QCB      ___ Control    × Warning  ___ AVG
```

Sodium

VEG7
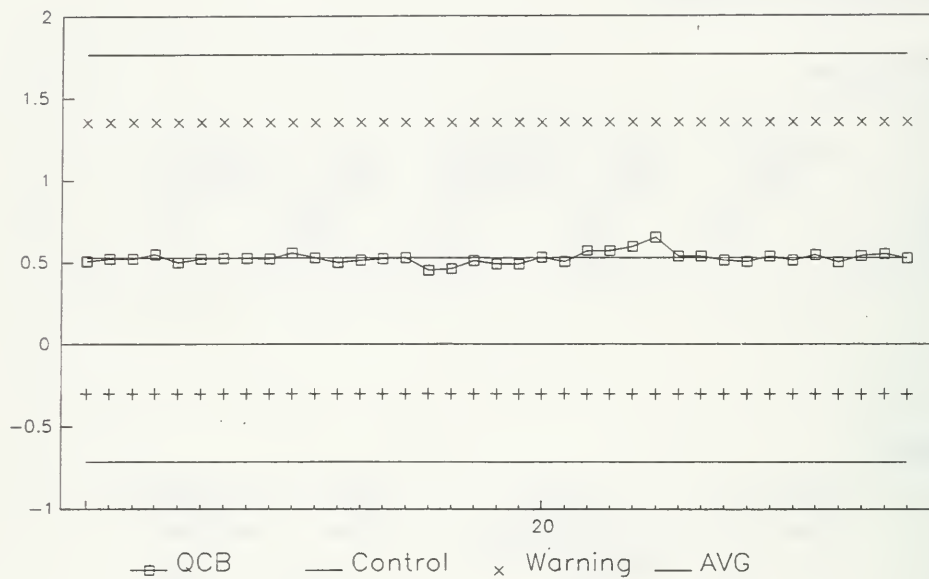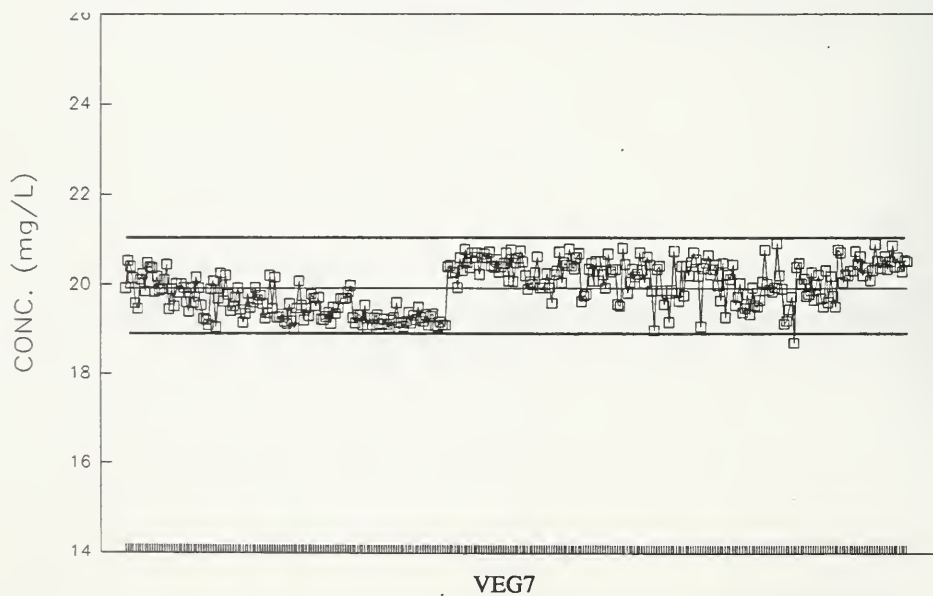
MERCURY

IDENTIFICATION:

Method Title: The Determination of Mercury in Water, Industrial Waste and Sewage by AAS.

Work Station Code:	TBHGW	Date Introduced:	1980
Method Code:	E6014A	Current Revision:	Nov. 1992
		Section:	Trace Contaminants

PARAMETERS:

Element	LIS Code	W (ug/L)	T (ug/L)
Mercury	HGUT	.01	.05

SAMPLE TYPE/MATRIX:

Domestic water, surface water, groundwater, industrial waste, landfill test wells, sewage, etc.

ANALYTICAL PROCEDURE:

Mercury in the sample is converted to the inorganic (oxidized) form by acid digestion. The mercury is then reduced being stannous chloride and the concentration measured by cold vapour atomic absorption spectroscopy.

INSTRUMENTATION:

Pharmacia Model 100M Mercury Monitor; Linear, five point calibration.

QUALITY ASSURANCE:

Controls: QCA, QCB

Ref. Materials: EPA1, EPA287

Drift: Standards reanalyzed every 20 samples

Duplicates: 1 per 20 samples

Interlabs: MOEE Blind Audit Program

Reporting: Units: ug/L (ppb)
                  Sig. Figures: 2

MERCURY (TOTAL) — HGUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.01 — 0.4 ug/L (ppb)

Calibration Control:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCA:	40	0.300	0.293	97.7	3.1
QCB:	40	0.100	0.100	99.5	4.1
QCA+QCB:	40	0.400	0.393	98.1	2.7
QCA−QCB:	40	0.200	0.194	96.8	4.6

For 1993 Control Limits:

$$Sw (A-B) = \quad 0.0104$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
40	0.01	0.08	0.0066	0.0068
N/A	0.08	0.2	—	—
N/A	0.2	0.4	—	—

Detection Limit (DL) = | 0.01 ug/L |

Mercury
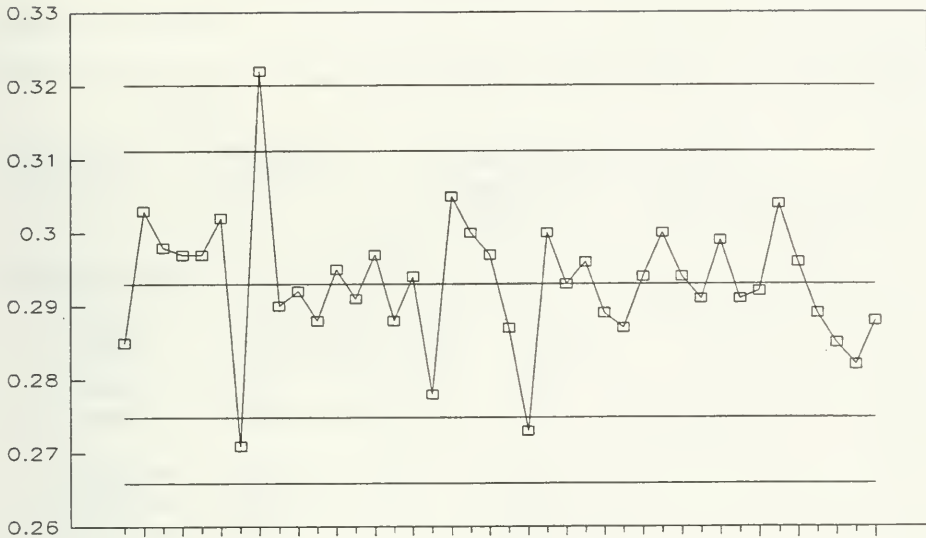
QCA

Mercury
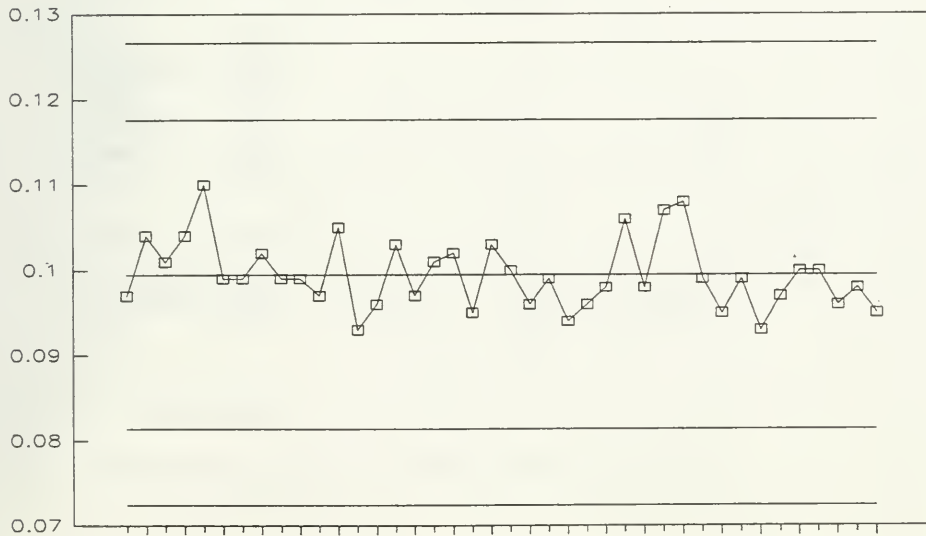
QCB

Mercury, QC Difference
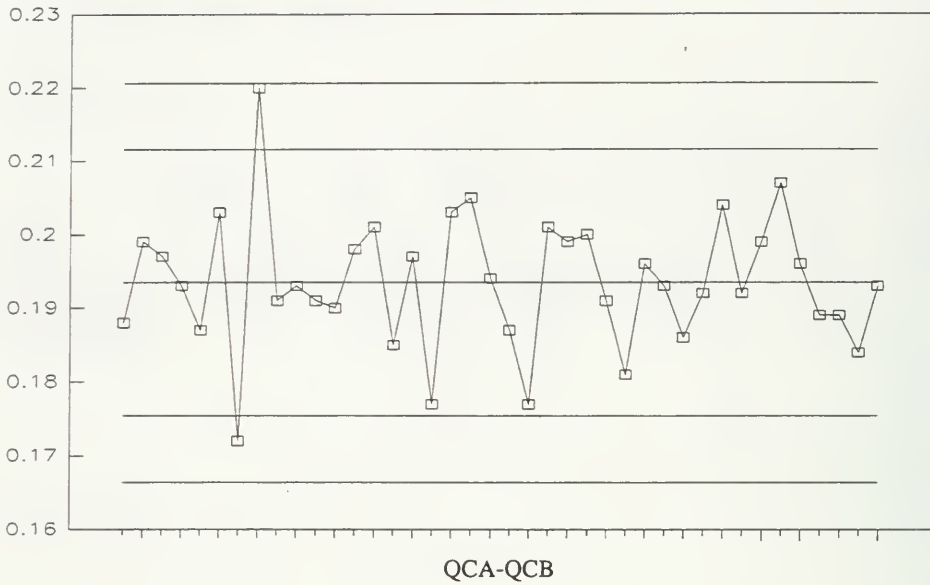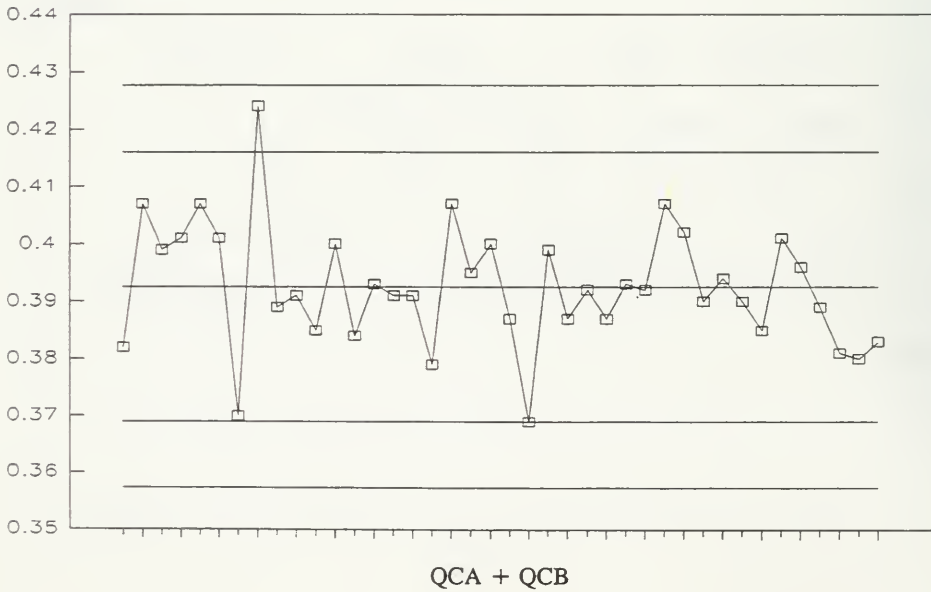
QCA-QCB

Mercury, QC Sum

QCA + QCB

TRACE METALS BY PRECONCENTRATION

IDENTIFICATION:

Method Title: The Determination of Trace Metals in Surface Waters by Preconcentration and ICP-AES.

WorkStation:	TBMPRE	Method Introduced:	May 1988
Method Code:	E6043A	Current Revision:	July 1993
		Section:	Trace Contaminants

PARAMETERS:

Element	LIS Code	W (ug/L)	T (ug/L)
Yttrium	YYUT	0.1	0.5
Beryllium	BEUT	0.1	0.5
Cadmium	CDUT	0.2	1.0
Chromium	CRUT	0.2	1.0
Molybdenum	MOUT	0.2	1.0
Cobalt	COUT	0.5	2.5
Copper	CUUT	0.5	2.5
Silver	AGUT	0.5	2.5
Vanadium	VVUT	0.5	2.5
Barium	BAUT	1	5
Manganese	MNUT	1	5
Nickel	NIUT	1	5
Strontium	SRUT	1	5
Titanium	TIUT	1	5
Zinc	ZNUT	1	5
Lead	PBUT	2	10
Aluminium	ALUT	5	25
Iron	FEUT	5	25

SAMPLE TYPE/MATRIX:

'Clean' samples such as drinking water, surface water and groundwater.

ANALYTICAL PROCEDURE:

Samples are subjected to nitric acid digestion/preconcentration followed by analysis by ICP-AES.

INSTRUMENTATION:

Thermo Jarrell Ash (ICAP61) Inductively Coupled Plasma Spectrometer; Linear, two-point calibration.

QUALITY ASSURANCE:

Controls:	Blank, QCC, QCD, QCE (Ag only)
Ref. Materials:	ERA PP/CLP Trace Metals Standard EPA ICAP 7 EPA ICAP 19
Drift:	QCD, QCE analyzed every 20 samples
Duplicates:	1 per 20 samples
Interlabs:	MOEE Blind Audit Program (Bimonthly) Great Lakes Action Program(GLAP, 2x annually) CAEAL Certification Program (2x annually)
Reporting:	Units: ug/L (ppb) Sig. Figures: 2
REMARKS:	See Section 1.2 for parameters that are accredited with CAEAL.

Analytical Range: 5 — 50,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	10.000	9.828	98.3	2.2
QCD:	192	2.000	1.958	97.9	4.2
QCC+QCD:	192	12.000	11.785	98.2	2.3
QCC−QCD:	192	8.000	7.870	98.4	2.2

For 1994 Control Limits:

$$Sw\ (C-D) = \quad 0.176$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
145	5	500	72.00	4.2000
30	500	5000	1700.0	77.00
N/A	5000	50000	—	—

Detection Limit (DL) = | 5 ug/L |

Aluminum, QCC (TBMPRE)
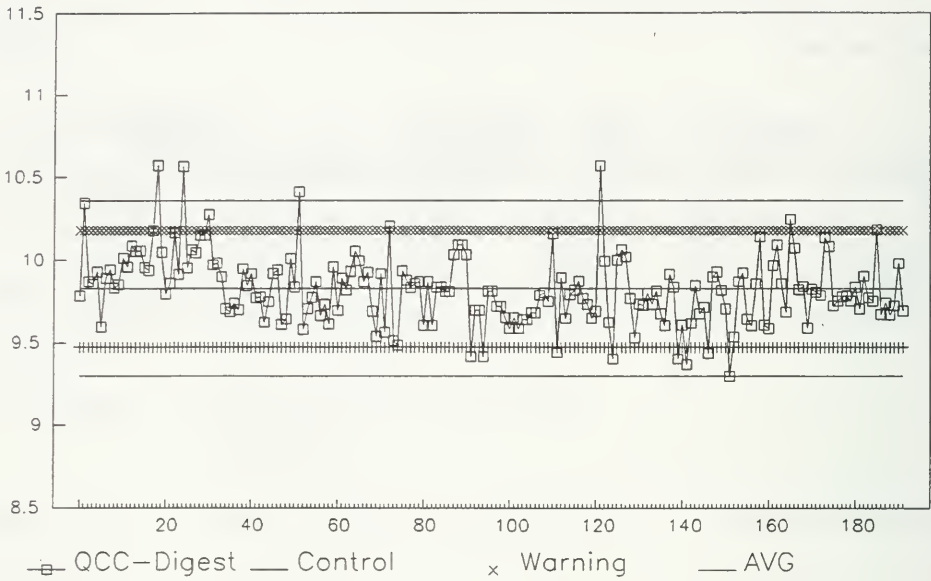
Aluminum, QCD (TBMPRE)
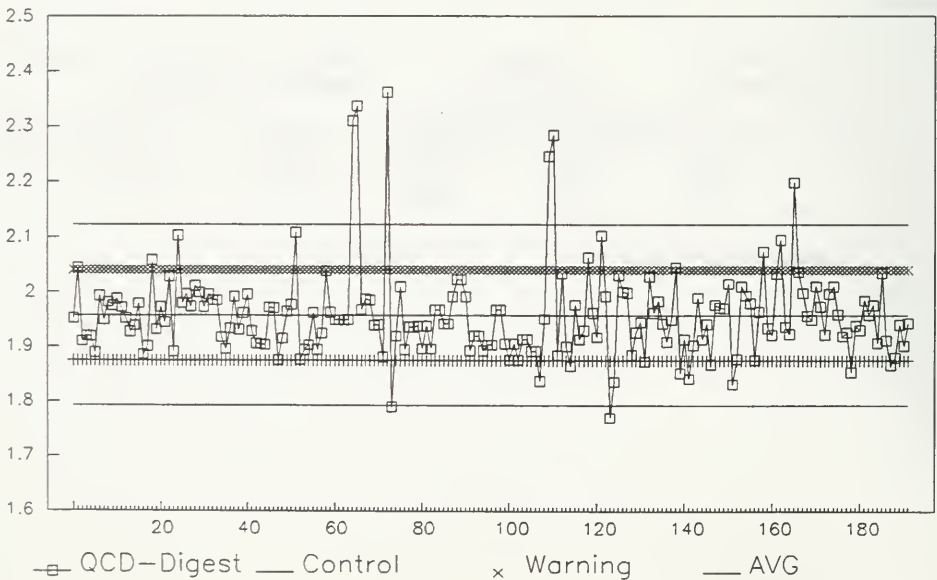
Aluminum, QC Difference (TBMPRE)
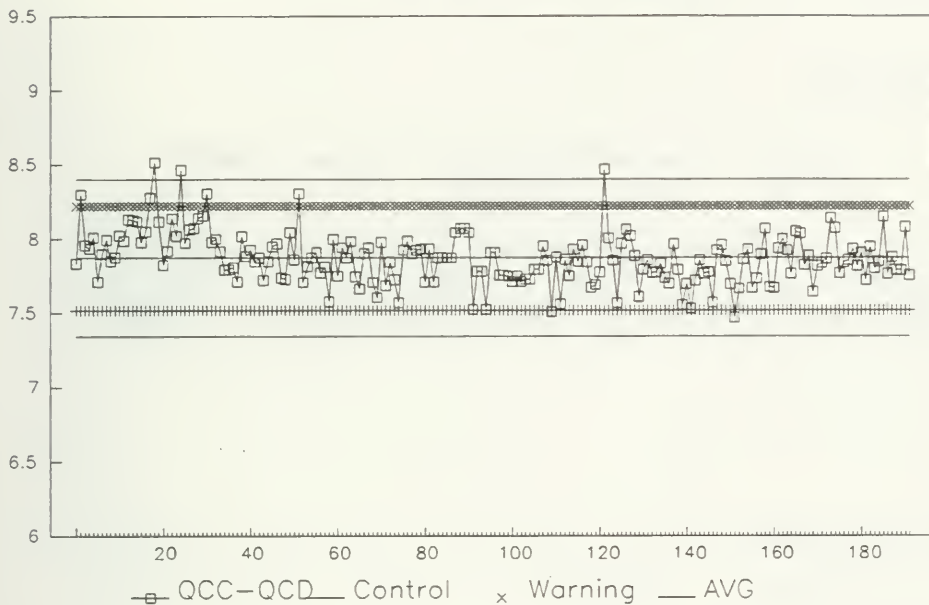
Aluminum, QC Sum (TBMPRE)
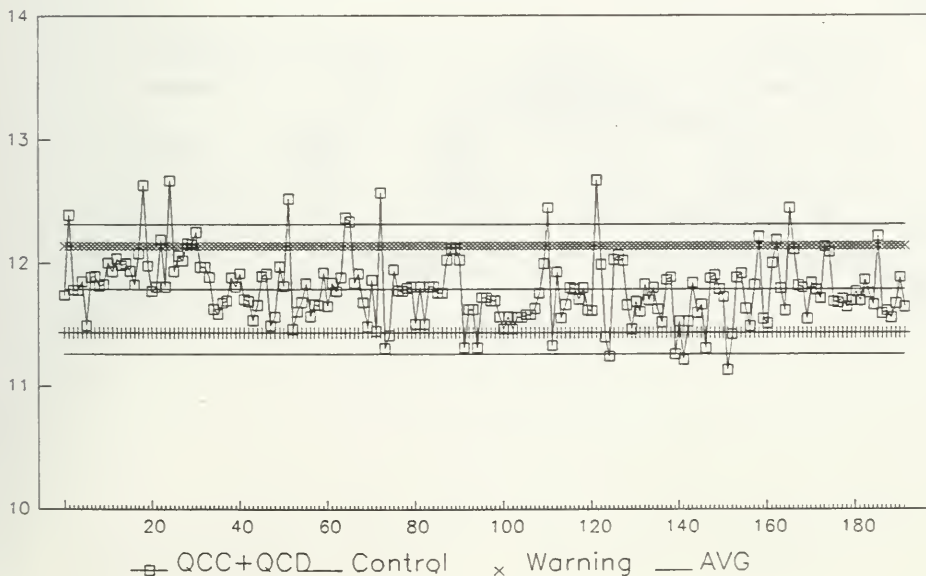
Analytical Range — 1–10,000 ug/L

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	Average Bias	Standard Deviation
QCC:	192	1.000	0.988	−0.012	2.6000
QCD:	192	0.200	0.198	−0.002	3.7000
QCC+QCD:	192	1.200	1.196	−0.004	2.6000
QCC−QCD:	192	0.800	0.800	0.000	2.9000

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0233$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
160	1	100	24.00	0.8400
17	100	1000	230.0	4.20
N/A	1000	10000	—	—

Detection Limit (DL) = 　　　　1 ug/L

Barium, QCC (TBMPRE)

Barium, QCD (TBMPRE)

197
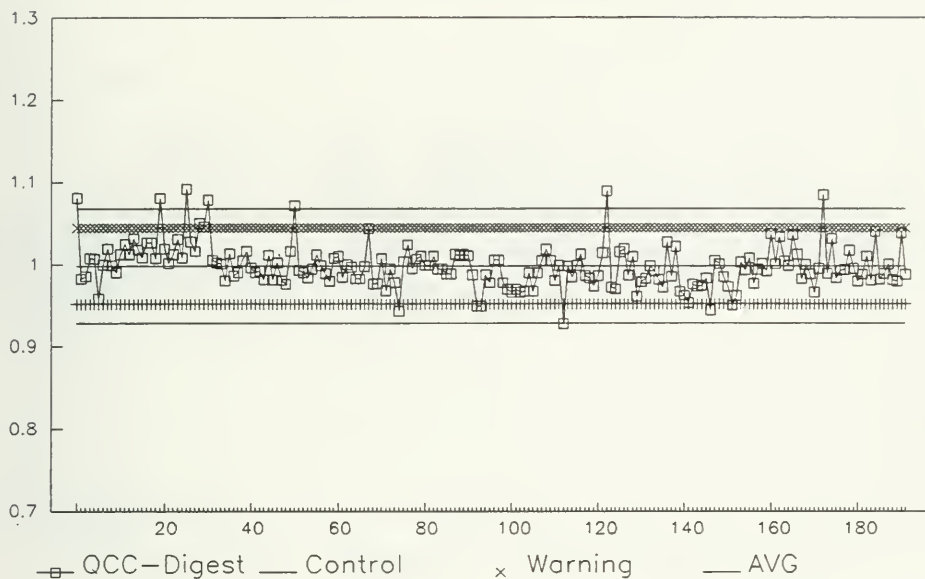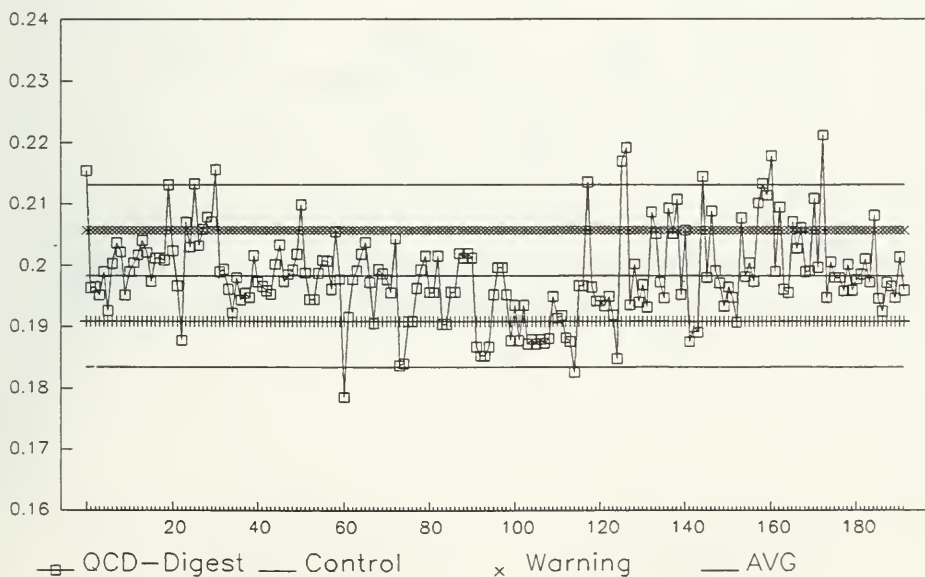
Barium, QC Difference (TBMPRE)
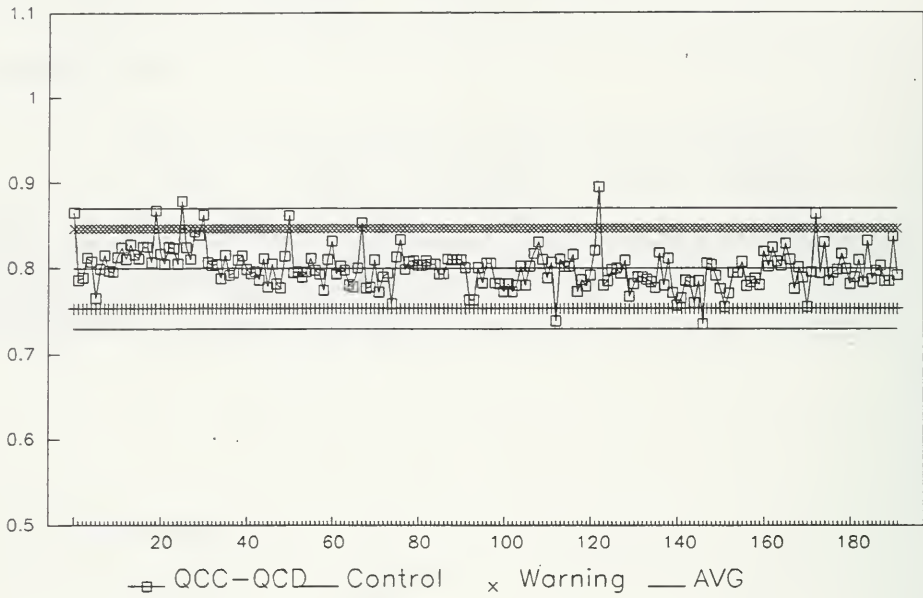
Barium, QC Sum (TBMPRE)
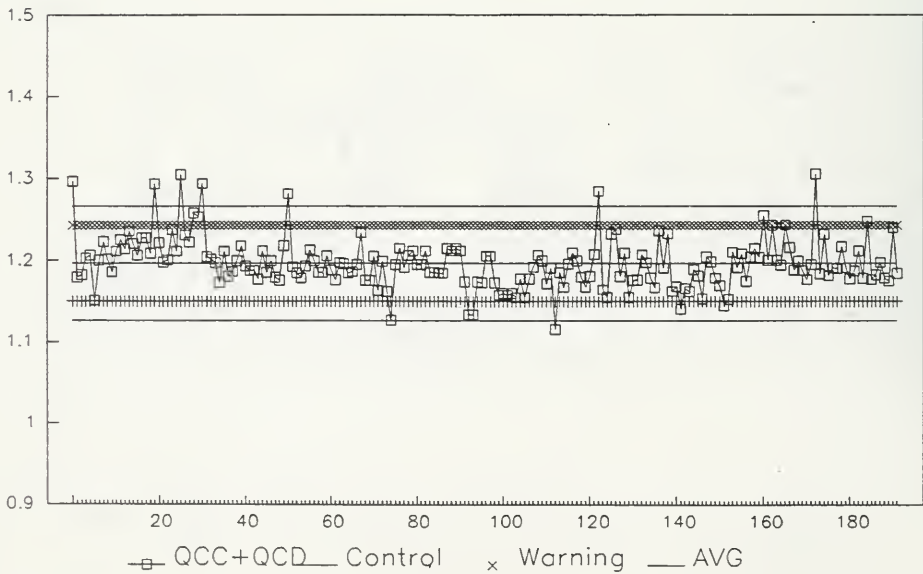
TBMPRE – BERYLLIUM (TOTAL) – BEUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.1 – 10,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.931	96.6	2.2
QCD:	192	0.400	0.379	94.8	2.3
QCC+QCD:	192	2.400	2.310	96.3	2.2
QCC−QCD:	192	1.600	1.553	97.1	2.2

For 1994 Control Limits:

$Sw (C-D) =$ 0.0344

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
180	0.1	10	0.29	0.051
N/A	10	100	–	–
N/A	100	1000	–	–

Detection Limit (DL) =   0.1 ug/L

Beryllium, QCC (TBMPRE)
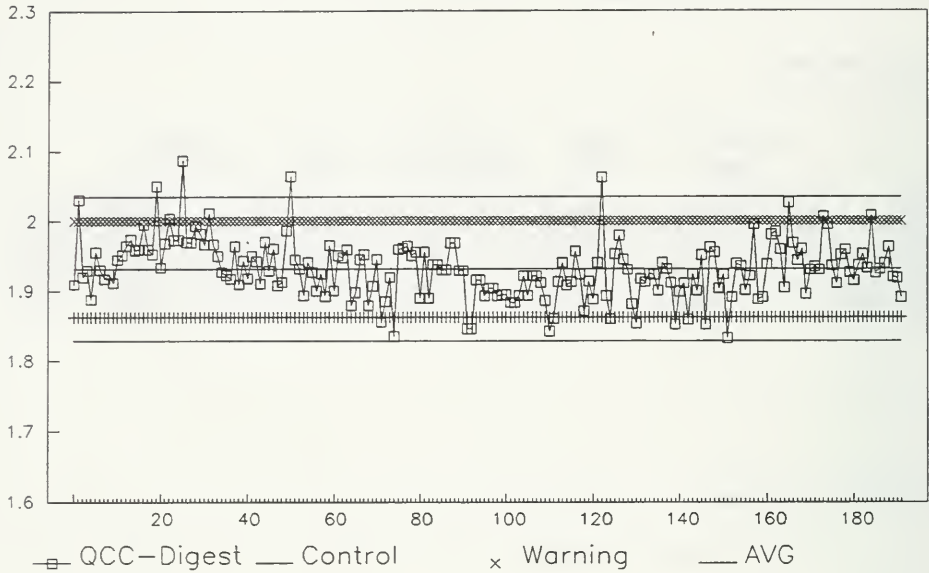
Beryllium, QCD (TBMPRE)
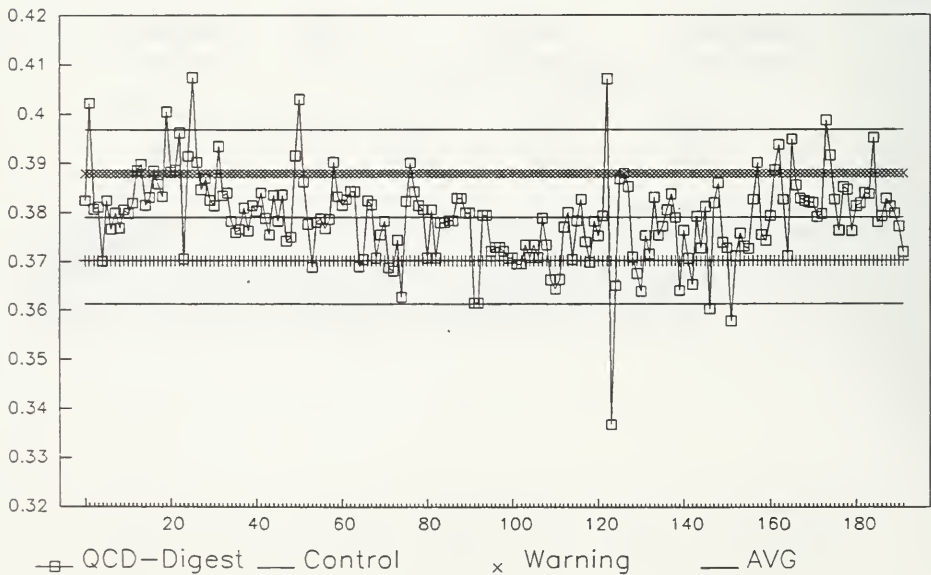
Beryllium, QC Difference (TBMPRE)
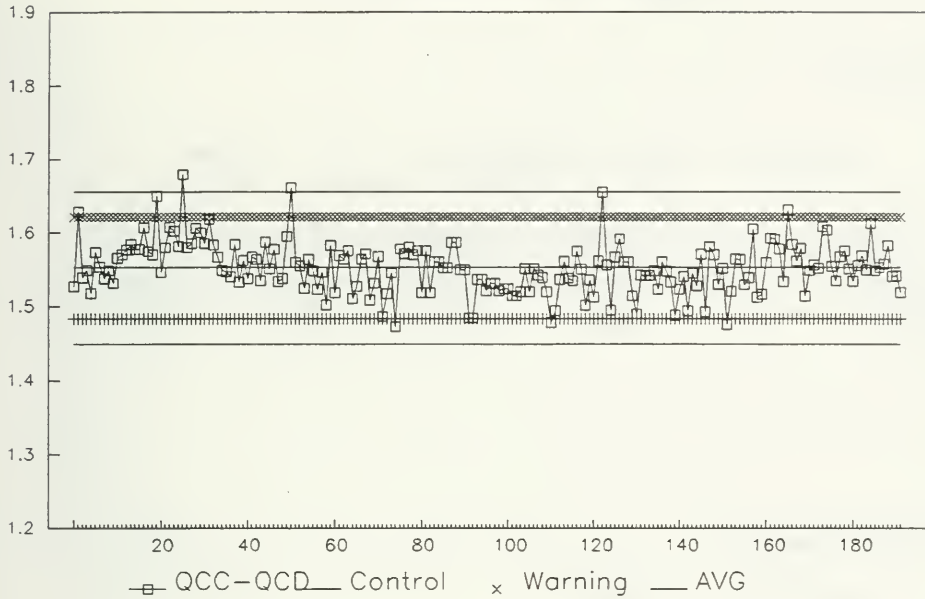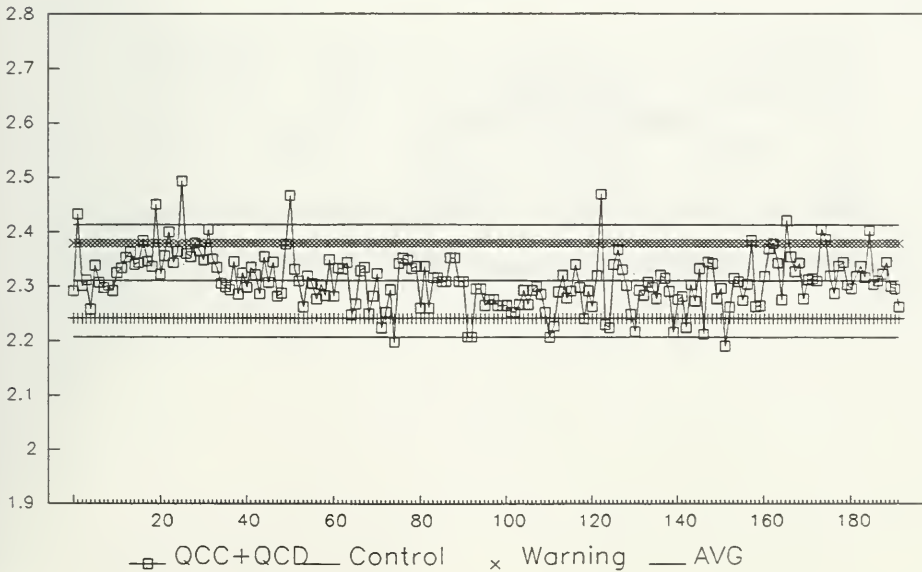

```
            QCC–QCD__ Control    × Warning ___ AVG
```

Beryllium, QC Sum (TBMPRE)


```
            QCC+QCD__ Control    × Warning ___ AVG
```

Analytical Range: 0.2 — 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	193	1.000	0.983	98.3	2.3
QCD:	193	0.200	0.195	97.5	3.0
QCC+QCD:	193	1.200	1.178	98.2	2.3
QCC−QCD:	193	0.800	0.788	98.5	2.4

For 1994 Control Limits:

$Sw (C-D) =$ 0.0187

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
176	0.2	20	0.34	0.0770
N/A	20	200	—	—
N/A	200	2000	—	—

Detection Limit (DL) =   0.2 ug/L

Cadmium, QCC (TBMPRE)
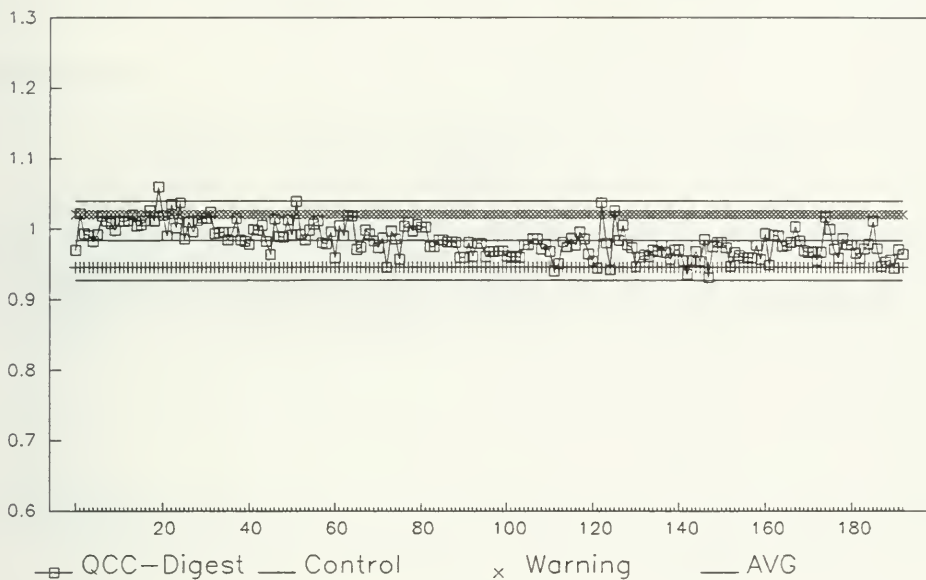
Cadmium, QCD (TBMPRE)
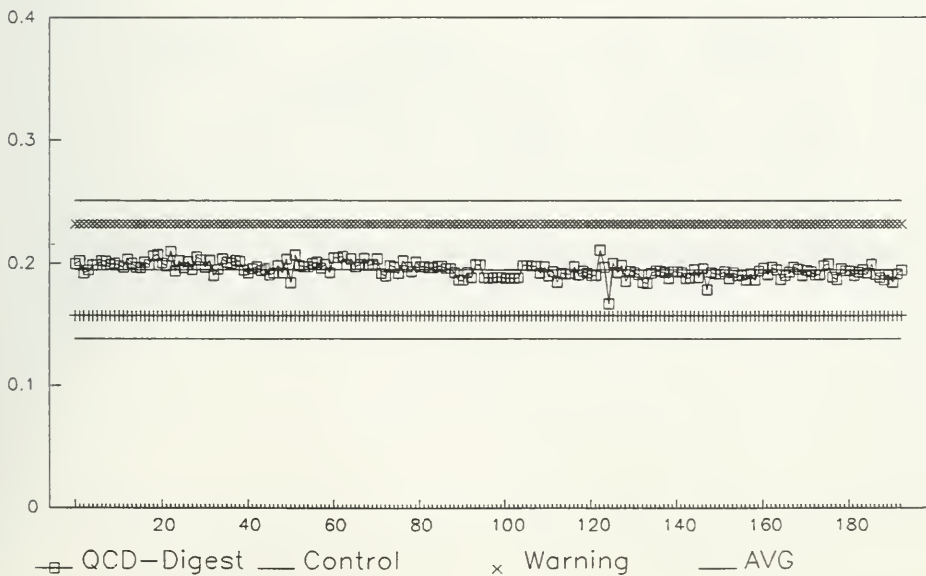
Cadmium, QC Difference (TBMPRE)
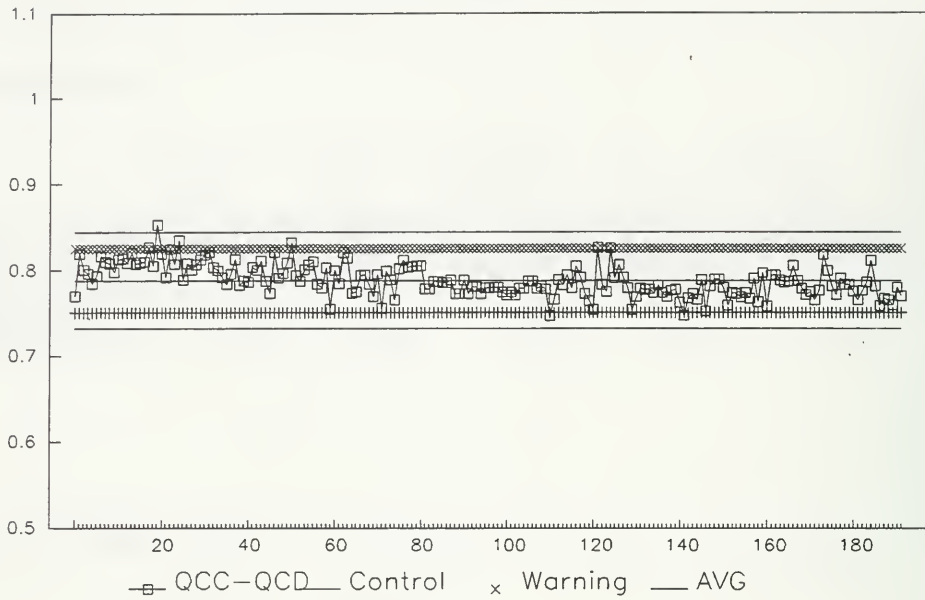
Cadmium, QC Sum (TBMPRE)
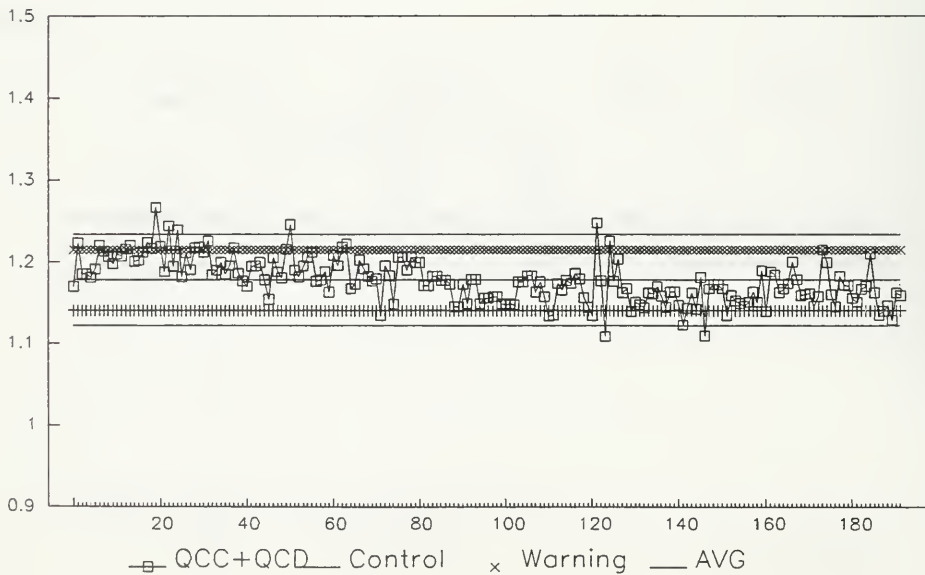
TBMPRE – CHROMIUM (TOTAL) – CRUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.2 – 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.917	95.9	2.1
QCD:	192	0.400	0.380	95.0	2.9
QCC+QCD:	192	2.400	2.297	95.7	2.1
QCC−QCD:	192	1.600	1.537	96.1	2.2

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.033$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
173	0.2	20	0.74	0.17
6	20	200	63.	1.50
N/A	200	2000	–	–

Detection Limit (DL) = $\boxed{\text{0.2 ug/L}}$

Chromium, QCC (TBMPRE)
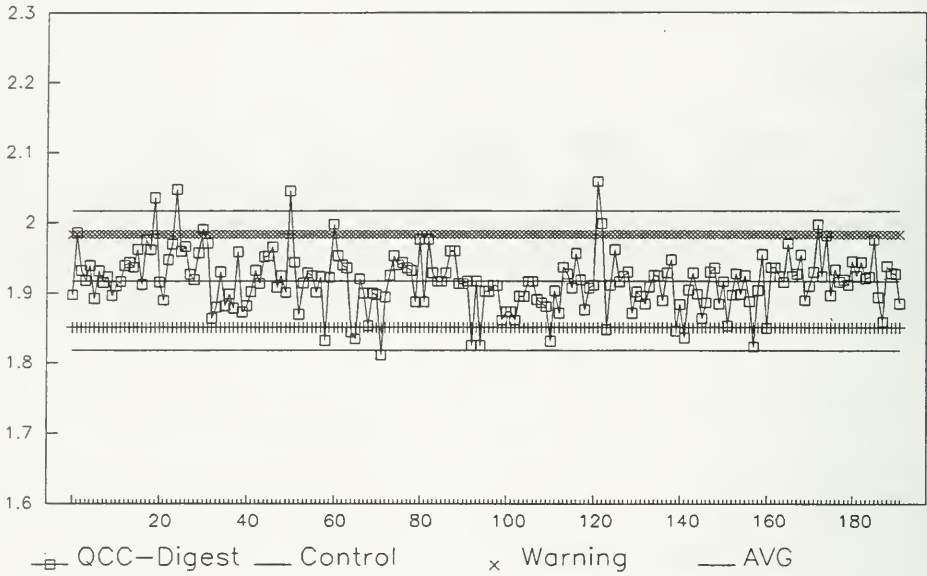
Chromium, QCD (TBMPRE)
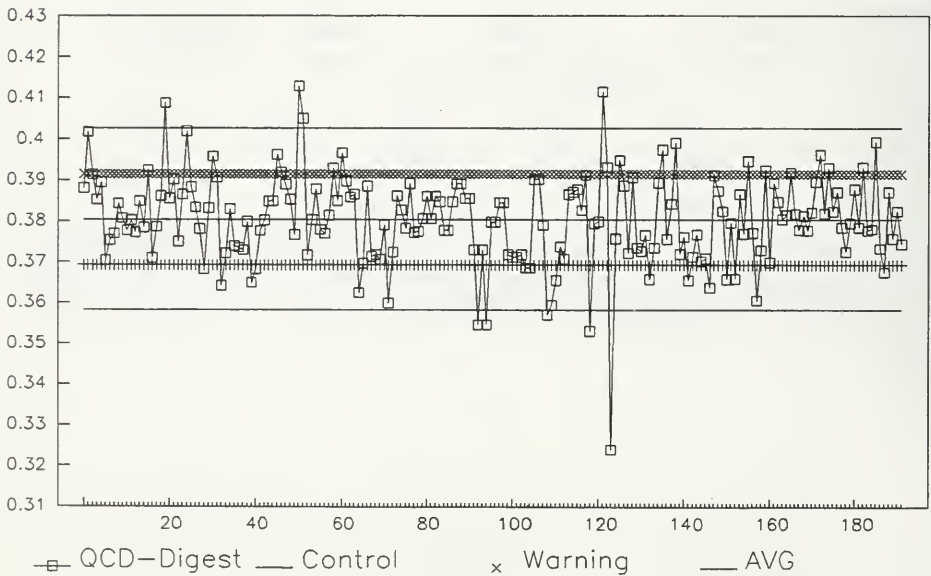
Chromium, QC Difference (TBMPRE)
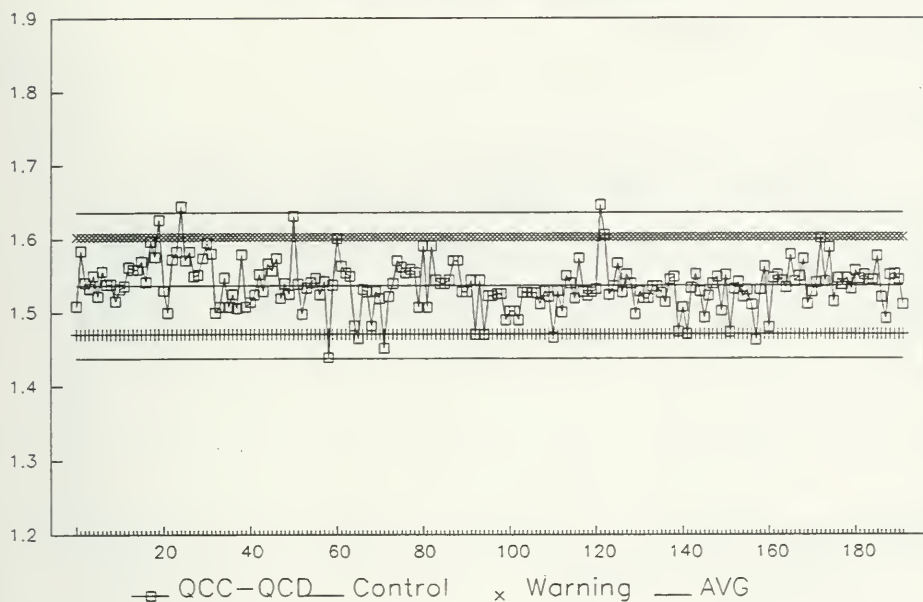
Chromium, QC Sum (TBMPRE)
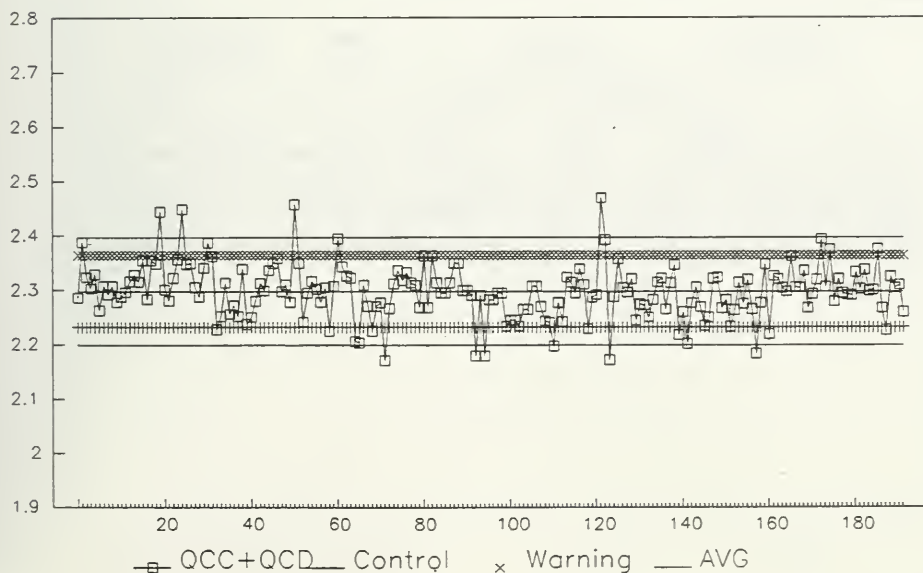
TBMPRE − COBALT (TOTAL) − COUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.5 − 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.991	99.6	2.1
QCD:	192	0.400	0.395	98.8	2.7
QCC+QCD:	192	2.400	2.386	99.4	2.1
QCC−QCD:	192	1.600	1.596	99.8	2.1

For 1994 Control Limits:

$$Sw (C-D) = \quad 0.0339$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
178	0.5	50	2.6	0.33
N/A	50	500	−	−
N/A	500	5000	−	−

Detection Limit (DL) = 0.5 ug/L

208
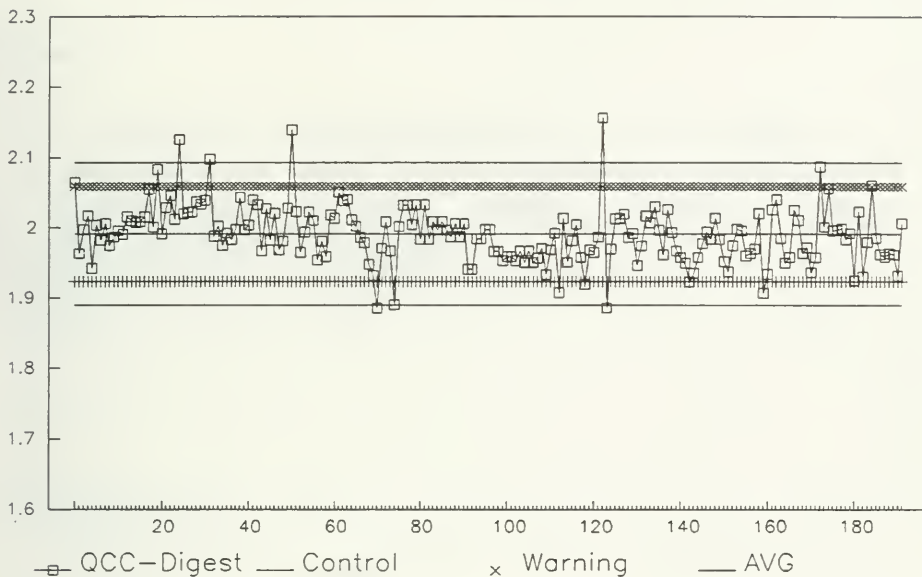
Cobalt, QCC (TBMPRE)

Cobalt, QCD (TBMPRE)
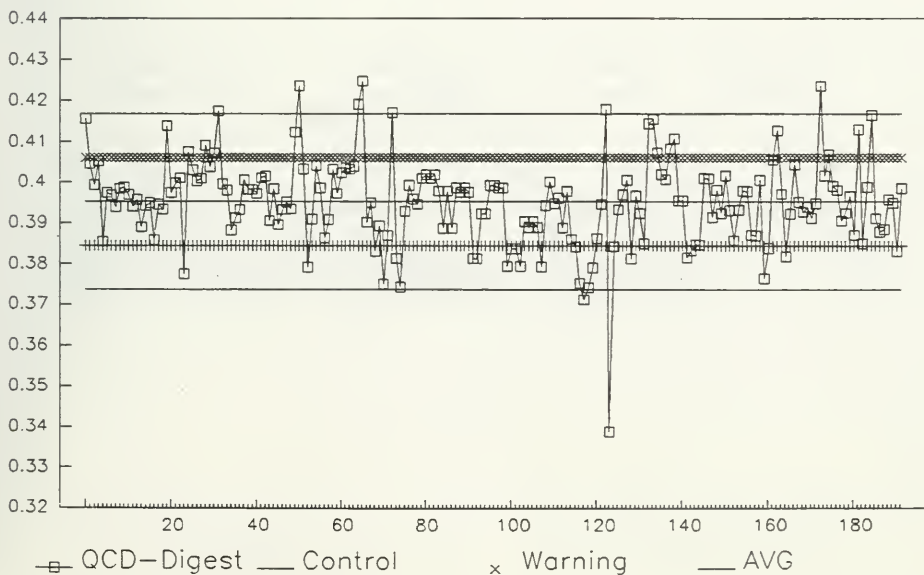
Cobalt, QC Difference (TBMPRE)
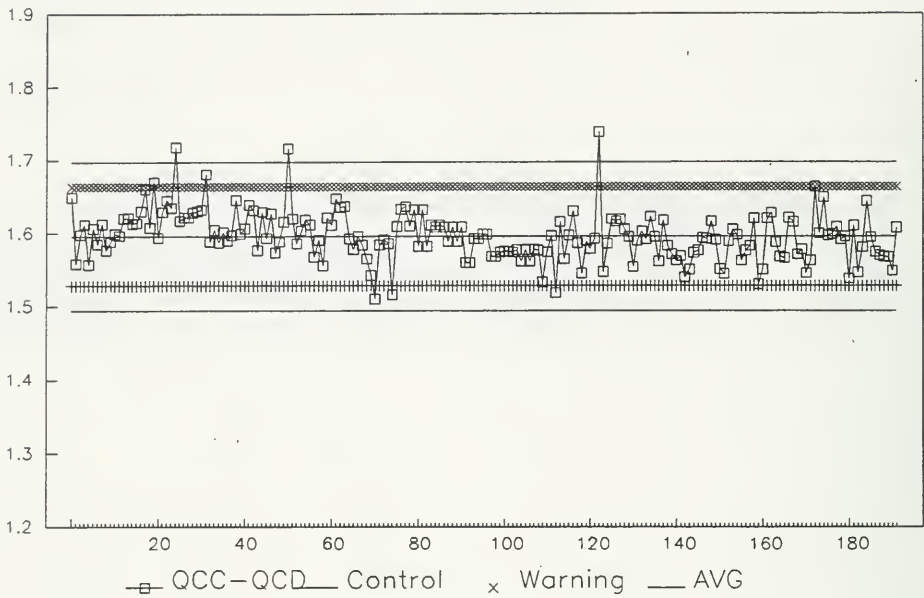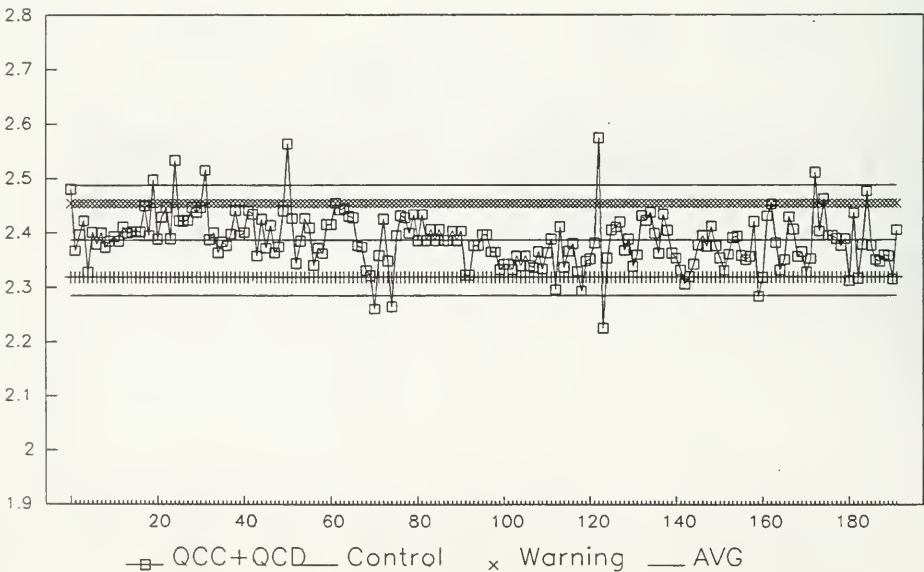
QCC–QCD _ Control   ×ₓ Warning __ AVG

Cobalt, QC Sum (TBMPRE)

QCC+QCD _ Control   ×ₓ Warning __ AVG

Analytical Range: 0.5 — 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.933	96.7	2.3
QCD:	192	0.400	0.386	96.5	5.5
QCC+QCD:	192	2.400	2.320	96.7	2.5
QCC−QCD:	192	1.600	1.547	96.7	2.4

For 1994 Control Limits:

$$Sw (C-D) = \quad 0.0369$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
148	0.5	50	5.90	0.2700
22	50	500	140.0	4.00
N/A	500	5000	—	—

Detection Limit (DL) =   0.5 ug/L

Copper, QCC (TBMPRE)

QCC-Digest ── Control ── Warning ── AVG

Copper, QCD (TBMPRE)

QCD-Digest ── Control ── Warning ── AVG
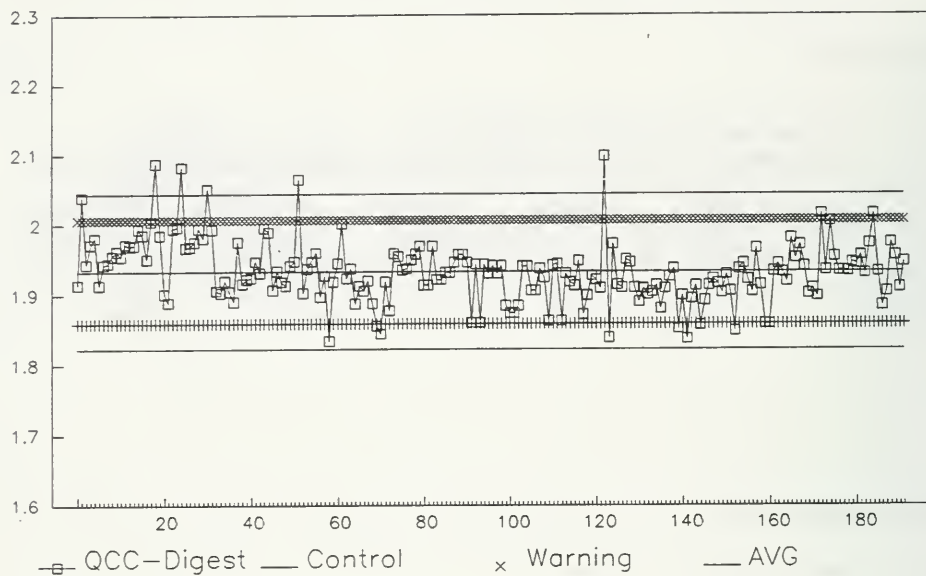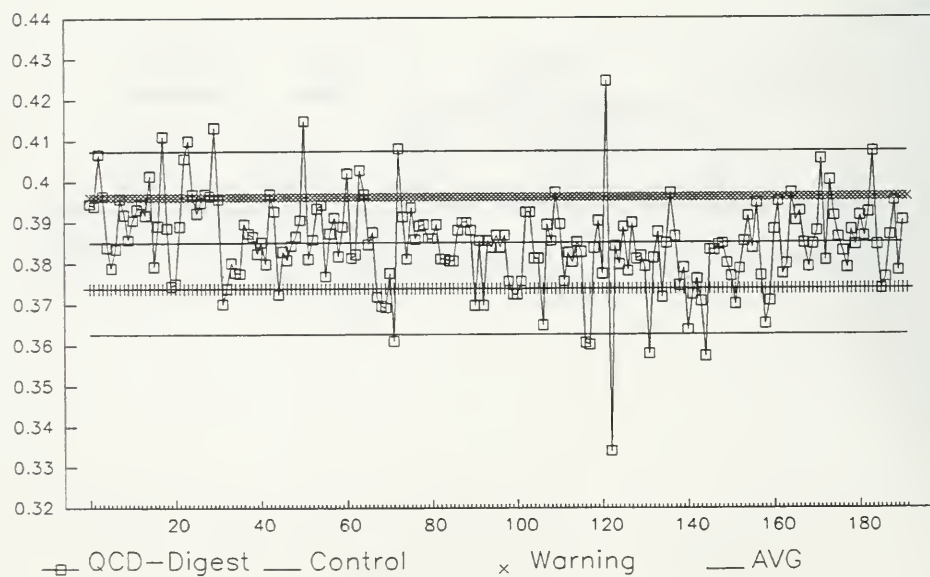
Copper, QC Difference (TBMPRE)
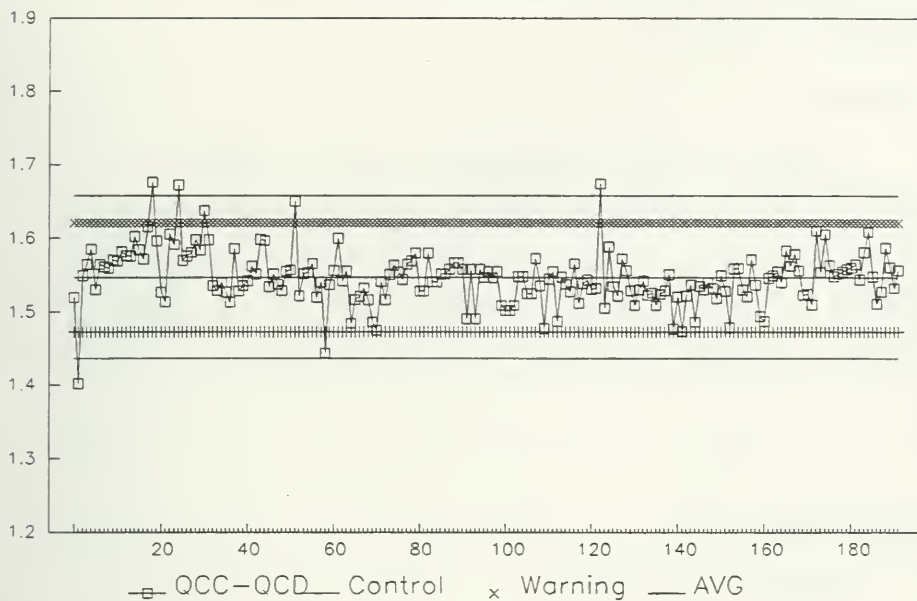
Copper, QC Sum (TBMPRE)
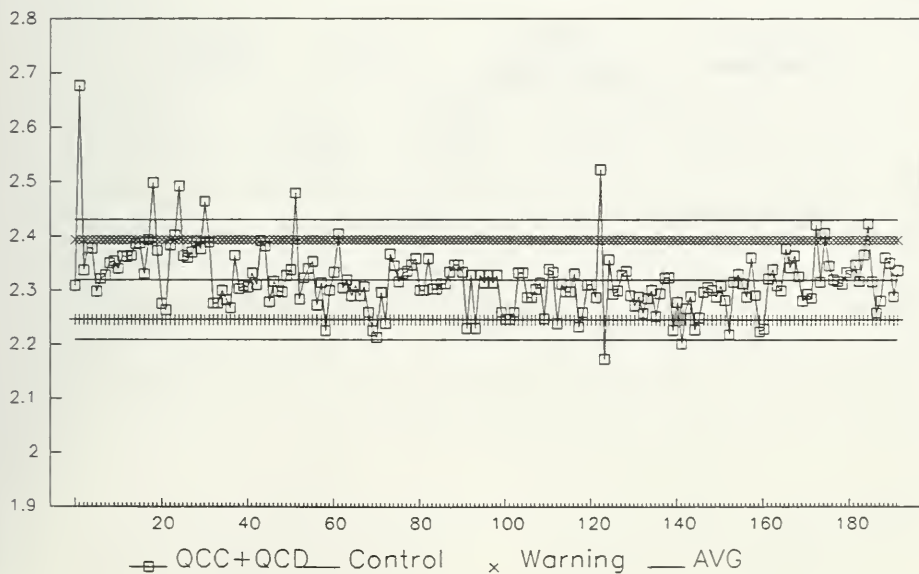
TBMPRE — IRON (TOTAL) — FEUT
Quality Control Data from January 1 to December 31, 1993

Analytical Range: 5 – 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	10.000	10.056	100.6	2.2
QCD:	192	2.000	2.008	100.4	4.8
QCC+QCD:	192	12.000	12.065	100.5	2.2
QCC−QCD:	192	8.000	8.049	100.6	2.6

For 1994 Control Limits:

$Sw (C-D) =$ 0.205

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
110	5	500	152	4.8
47	500	5000	1010	21.
8	5000	15000	6550	160.

Detection Limit (DL) = | 5 ug/L |

Iron, QCC (TBMPRE)
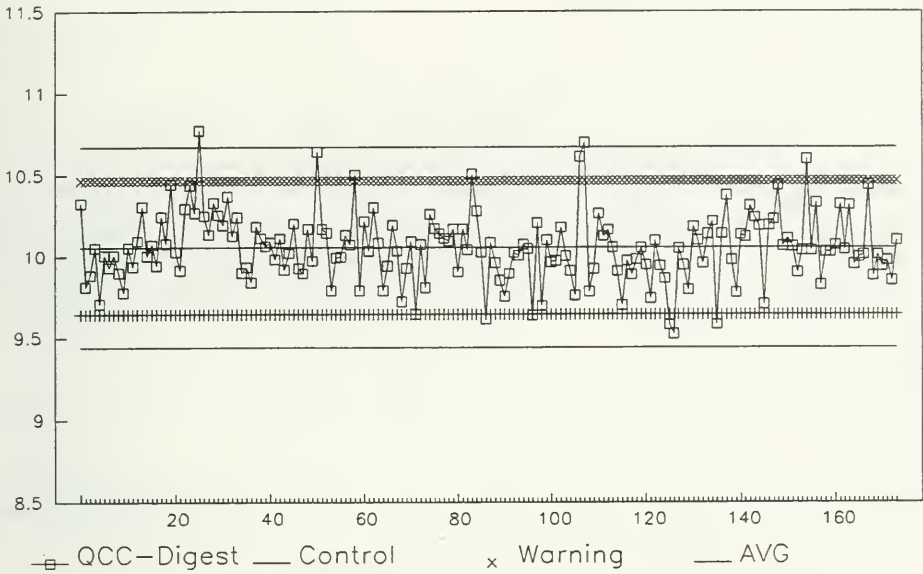
Iron, QCD (TBMPRE)
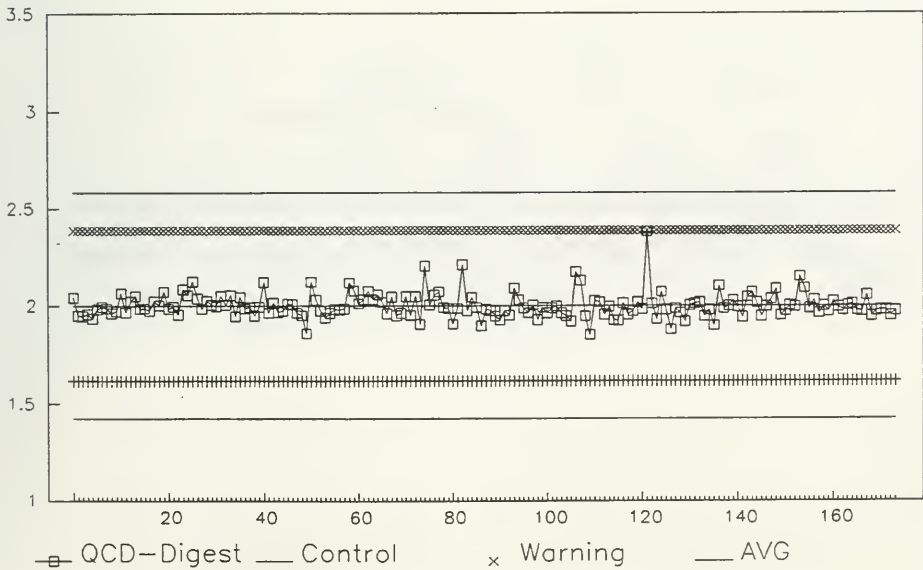
Iron, QC Difference (TBMPRE)
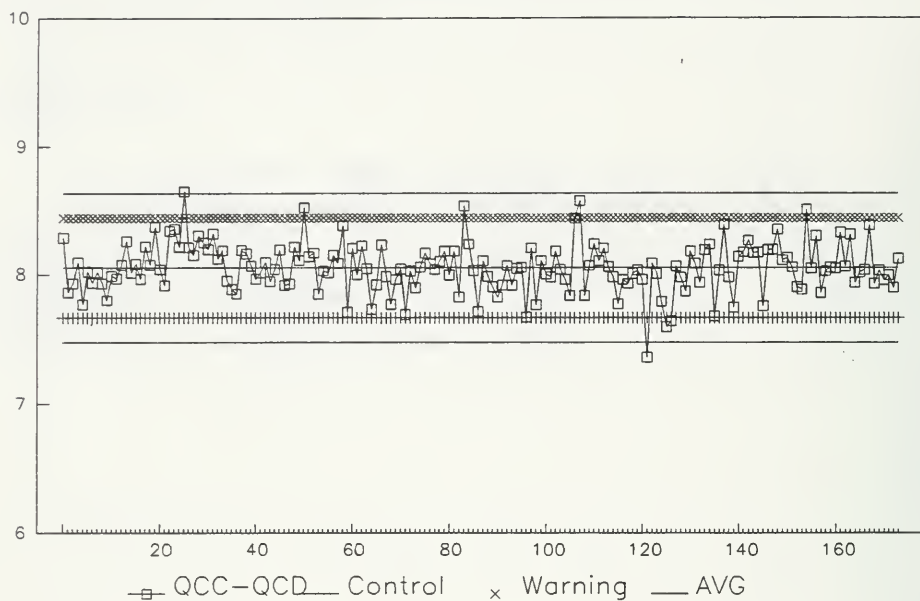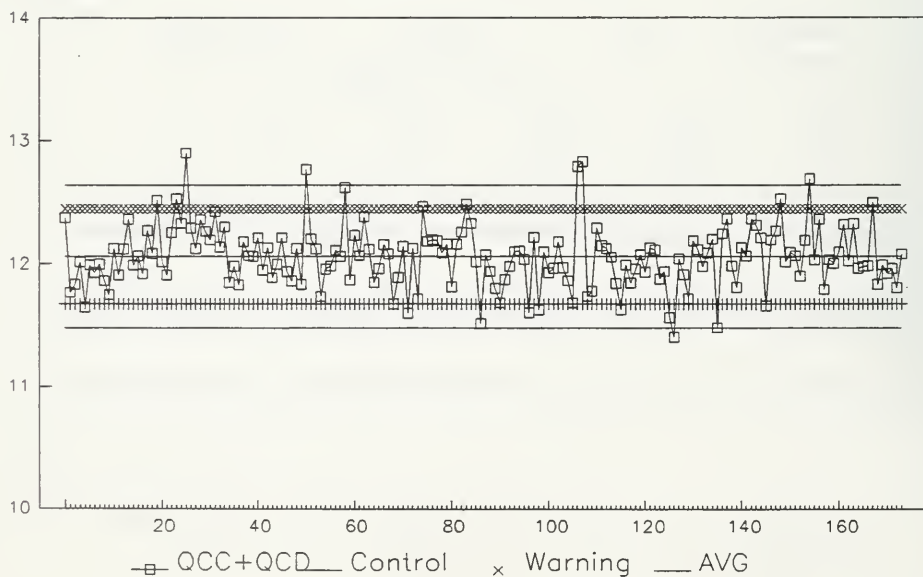
─□─ QCC−QCD ── Control   × Warning ── AVG

Iron, QC Sum (TBMPRE)

─□─ QCC+QCD ── Control   × Warning ── AVG

Analytical Range: 2 — 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.970	98.5	2.8
QCD:	192	0.400	0.396	99.0	9.5
QCC+QCD:	192	2.400	2.365	98.5	3.4
QCC−QCD:	192	1.600	1.574	98.4	3.1

For 1994 Control Limits:

$$Sw\ (C-D) = \quad 0.0498$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
174	2	200	6.00	0.9600
0	200	2000	—	—
0	2000	20000	—	—

Detection Limit (DL) = | 2 ug/L |

Lead, QCC (TBMPRE)
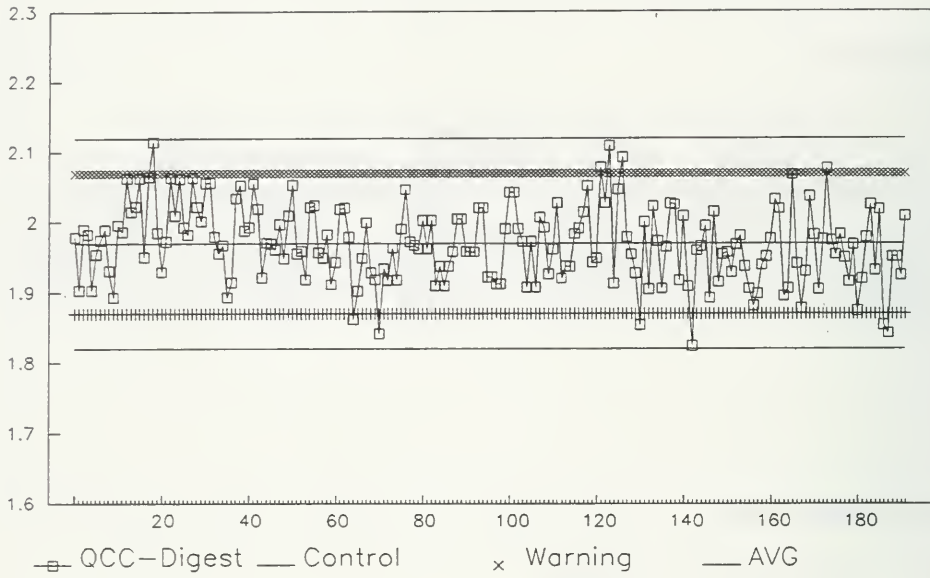
Lead, QCD (TBMPRE)
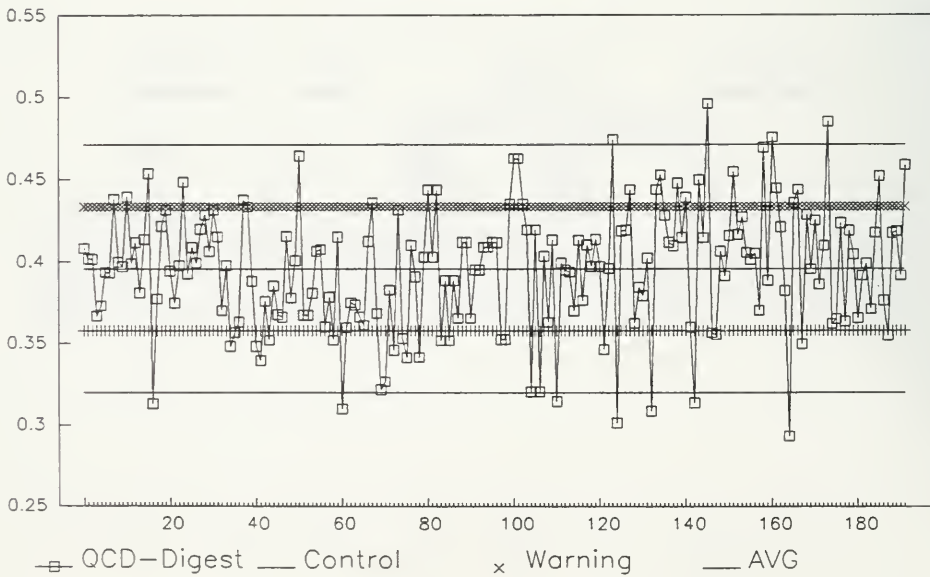
Lead, QC Difference (TBMPRE)
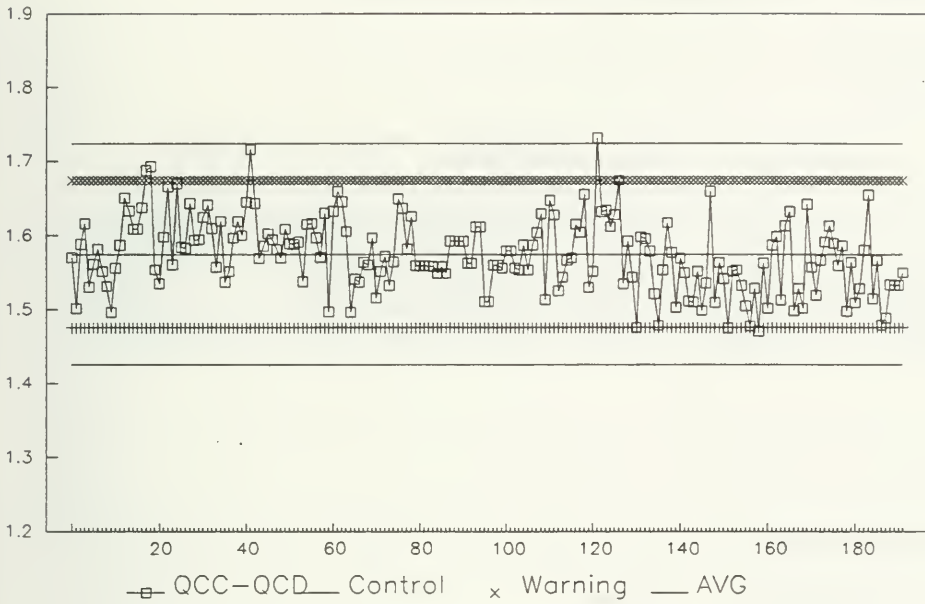
Lead, QC Sum (TBMPRE)
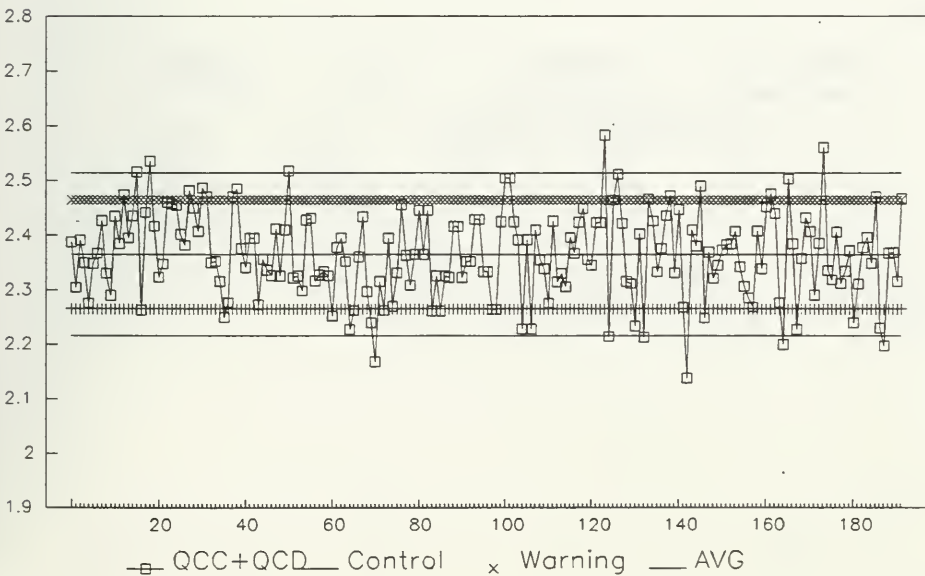
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Analytical Range: 1 — 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.957	97.9	2.4
QCD:	192	0.400	0.387	96.8	4.1
QCC+QCD:	192	2.400	2.344	97.7	2.4
QCC−QCD:	192	1.600	1.570	98.1	2.5

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0399$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
136	1	100	27.00	0.6300
35	100	1000	270.0	5.90
8	1000	10000	3390.00	160.0000

Detection Limit (DL) = | 1 ug/L |

Manganese, QCC (TBMPRE)

QCC-Digest ___ Control   x Warning   ___ AVG

Manganese, QCD (TBMPRE)

QCD-Digest ___ Control   x Warning   ___ AVG
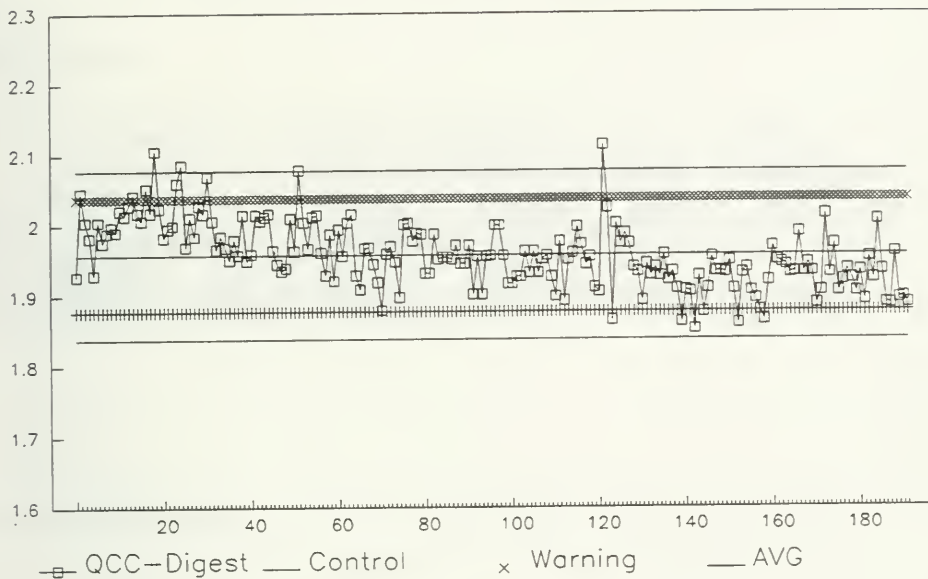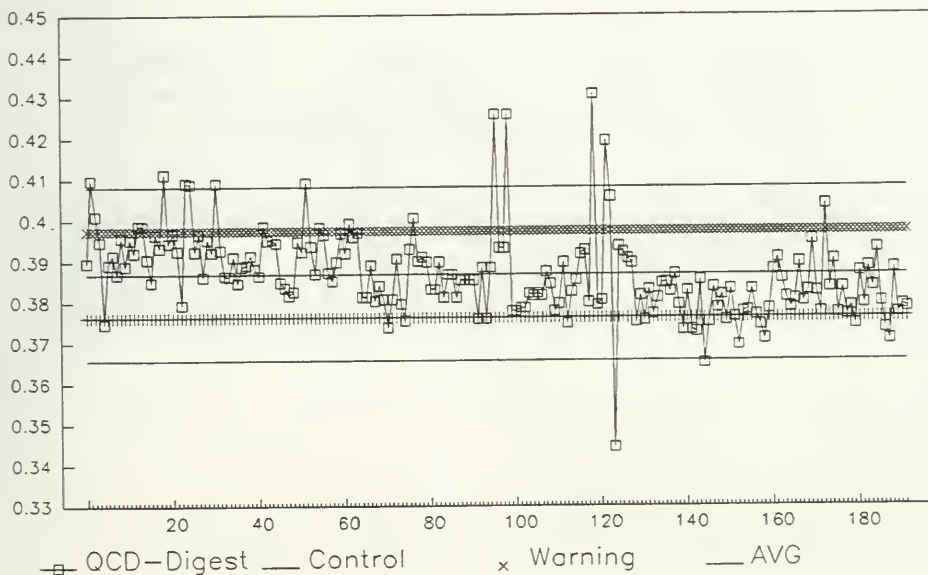
Manganese, QC Difference (TBMPRE)

Manganese, QC Sum (TBMPRE)
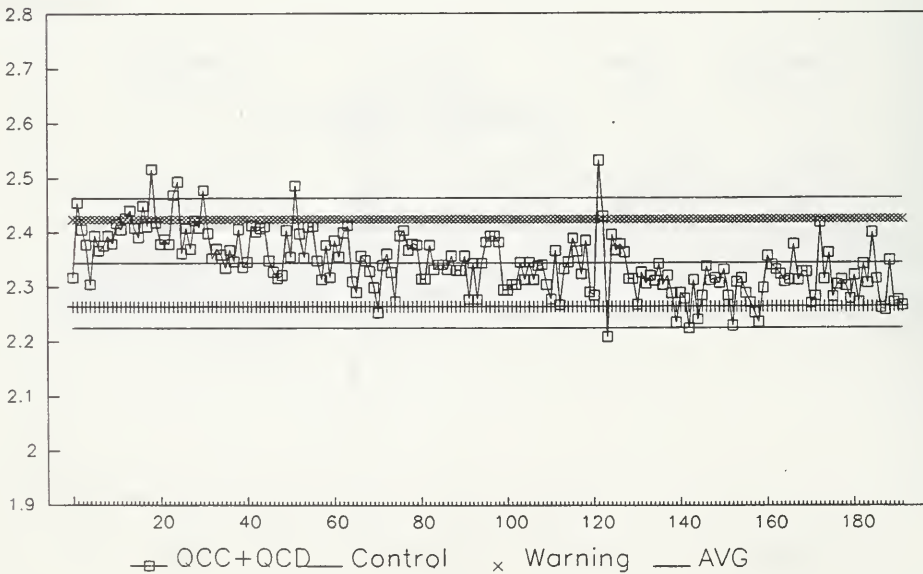
Analytical Range: 0.2 — 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.968	98.4	2.3
QCD:	192	0.400	0.387	96.8	2.9
QCC+QCD:	192	2.400	2.354	98.1	2.3
QCC−QCD:	192	1.600	1.581	98.8	2.4

For 1994 Control Limits:

$$Sw\,(C{-}D) = \quad 0.0373$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
174	0.2	20	1.10	0.1600
N/A	20	200	—	—
N/A	200	2000	—	—

Detection Limit (DL) = 0.2 ug/L

Molybdenum, QCC (TBMPRE)
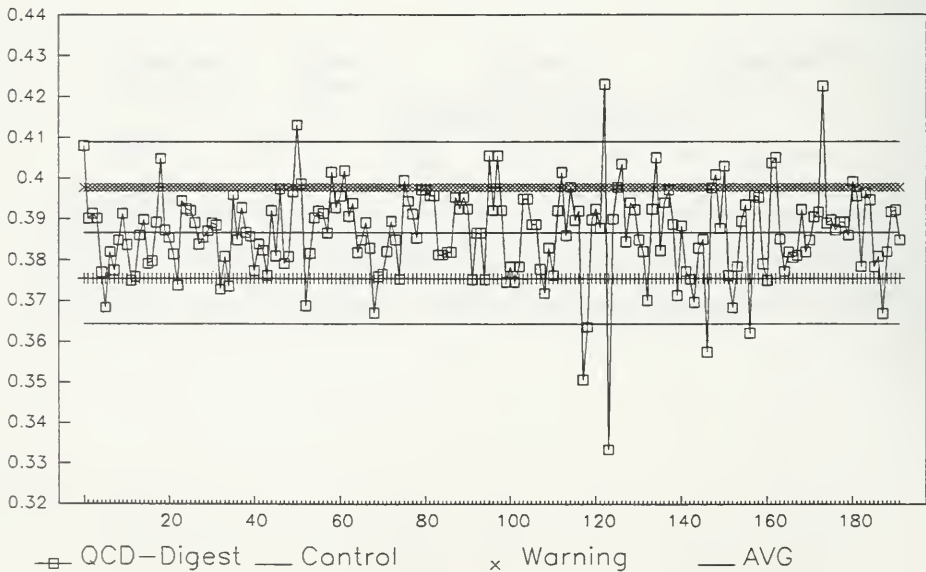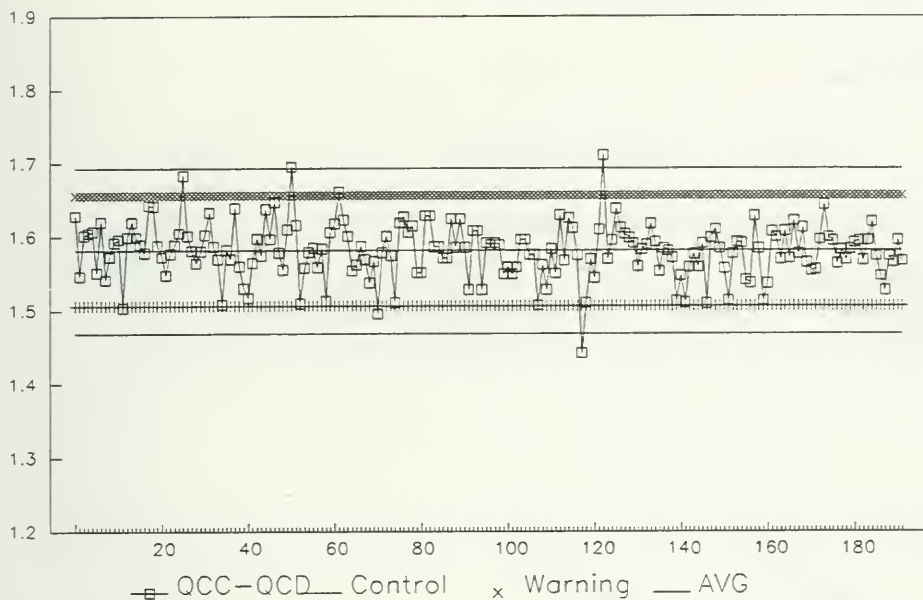
QCC–Digest ▭   Control ——   Warning ×   AVG ——

Molybdenum, QCD (TBMPRE)

QCD–Digest ▭   Control ——   Warning ×   AVG ——

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Molybdenum, QC Difference (TBMPRE)

QCC-QCD ___ Control   × Warning ___ AVG

Molybdenum, QC Sum (TBMPRE)

QCC+QCD ___ Control   × Warning ___ AVG
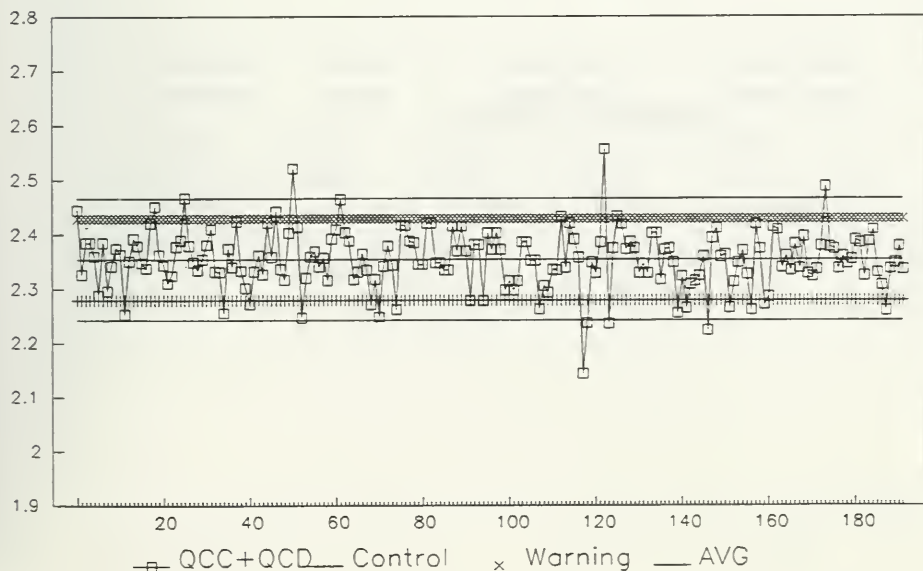
Analytical Range: 1 — 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.943	97.2	2.6
QCD:	192	0.400	0.386	96.5	3.3
QCC+QCD:	192	2.400	2.328	97.0	2.6
QCC−QCD:	192	1.600	1.557	97.3	2.7

For 1994 Control Limits:

Sw (C−D) = 0.042

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
168	1	100	3.50	0.5700
9	100	1000	236.0	4.20
N/A	1000	10000	—	—

Detection Limit (DL) = 1 ug/L

Nickel, QCC (TBMPRE)
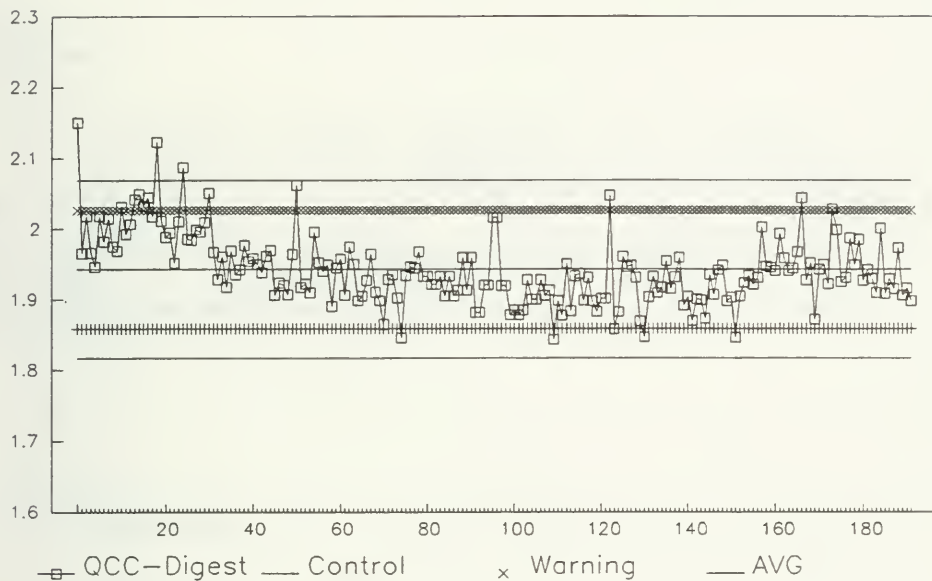
Nickel, QCD (TBMPRE)
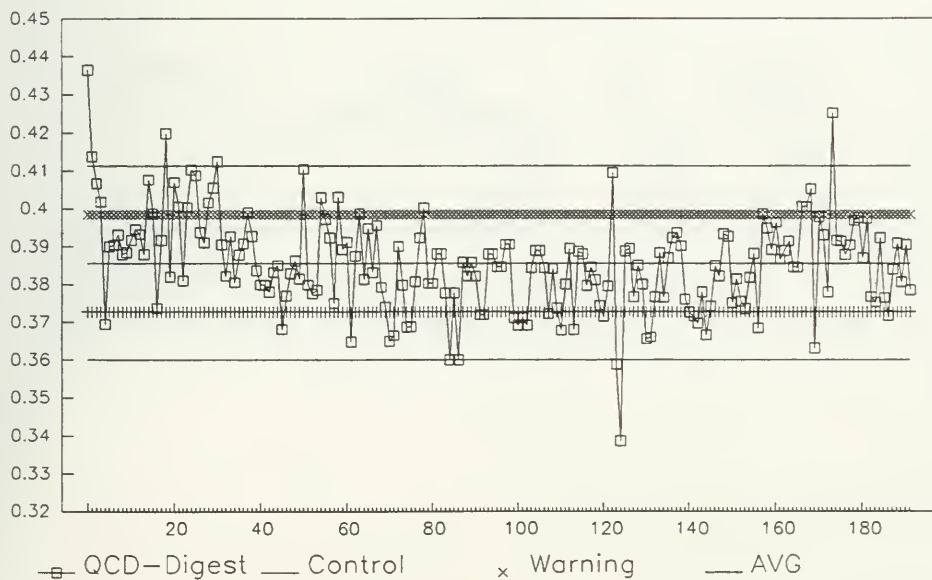
Nickel, QC Difference (TBMPRE)
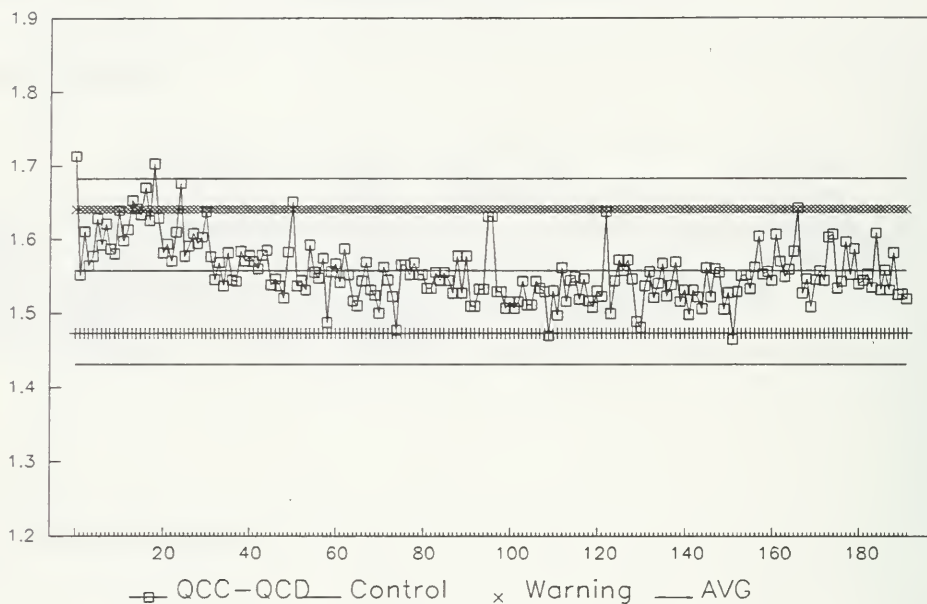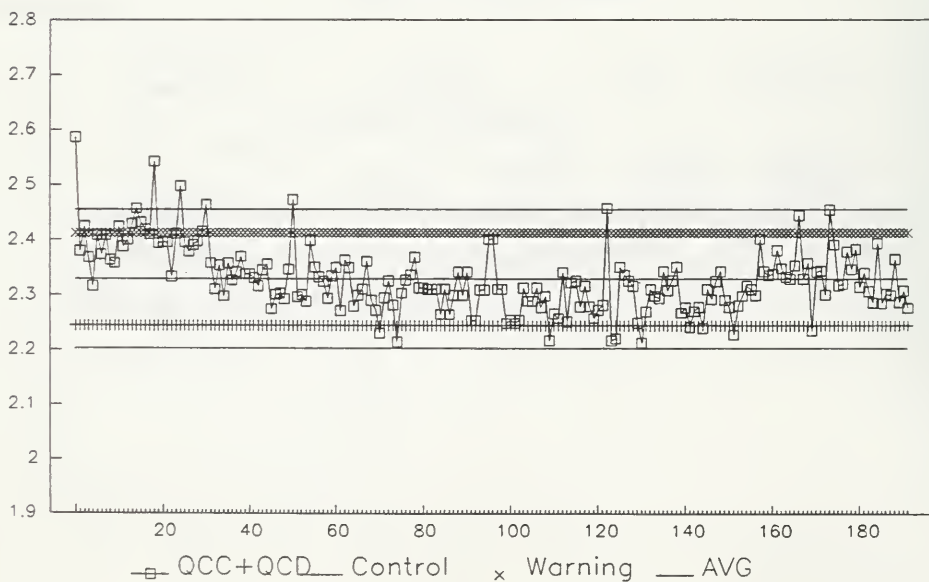
-□- QCC-QCD — Control ⨯ Warning — AVG

Nickel, QC Sum (TBMPRE)

-□- QCC+QCD — Control ⨯ Warning — AVG

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Analytical Range: 1 — 10,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	1.000	0.983	98.3	2.6
QCD:	192	0.200	0.195	97.5	3.2
QCC+QCD:	192	1.200	1.178	98.2	2.5
QCC−QCD:	192	0.800	0.789	98.6	2.7

For 1994 Control Limits:

$$Sw\,(C{-}D) = \quad 0.0216$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
117	1	100	38.00	0.9200
50	100	1000	300.0	6.20
10	1000	10000	2060.00	42.0000

Detection Limit (DL) = 1 ug/L

Strontium, QCC (TBMPRE)
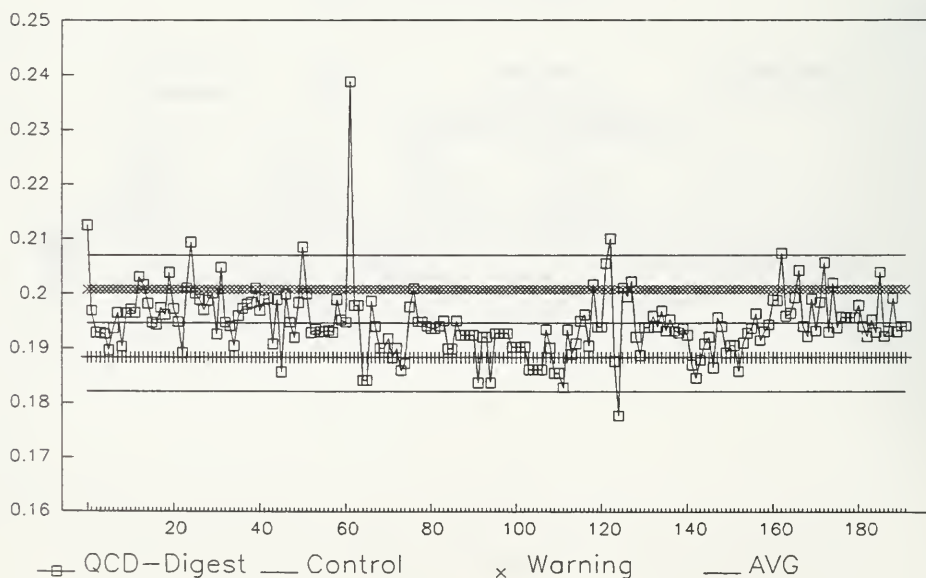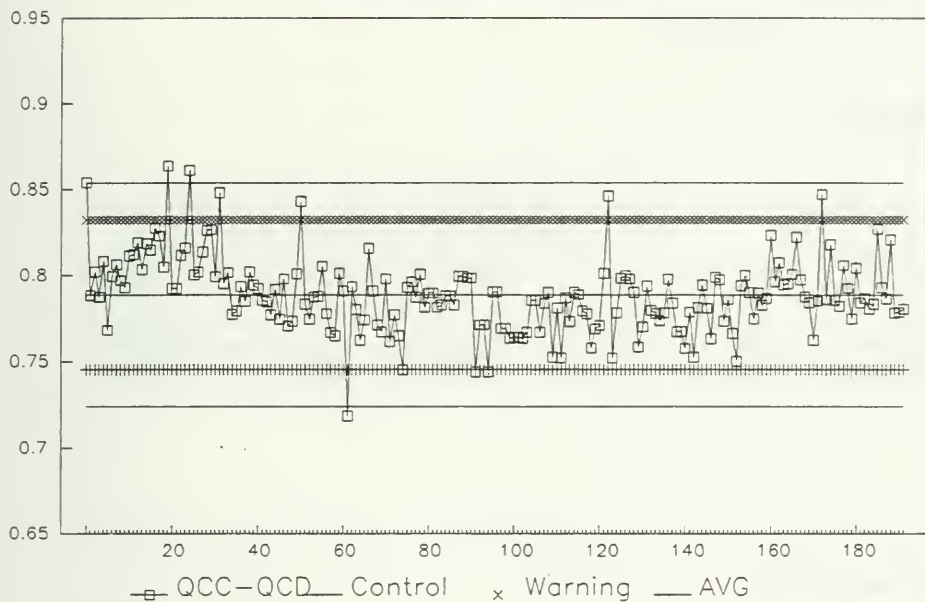
QCC—Digest ___ Control   ✕ Warning   ___ AVG

Strontium, QCD (TBMPRE)

QCD—Digest ___ Control   ✕ Warning   ___ AVG

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Strontium, QC Difference (TBMPRE)

_□_ QCC-QCD __ Control   × Warning ___ AVG

Strontium, QC Sum (TBMPRE)

_□_ QCC+QCD __ Control   × Warning ___ AVG
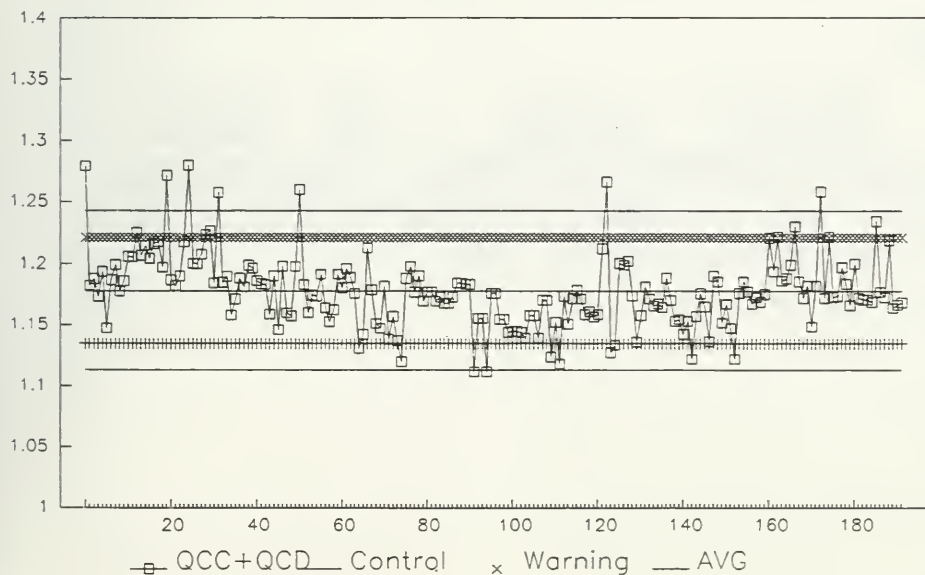
Analytical Range: 1 − 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.960	98.0	3.0
QCD:	192	0.400	0.375	93.8	3.9
QCC+QCD:	192	2.400	2.355	98.1	3.0
QCC−QCD:	192	1.600	1.584	99.0	3.1

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0488$$

Duplicates:

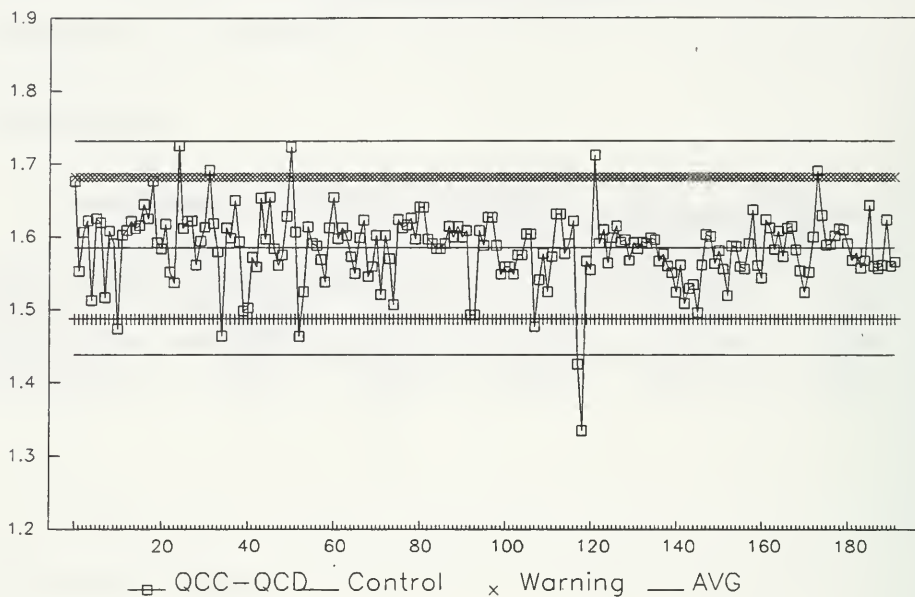| Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
166	1	100	6.70	0.6200
N/A	100	1000	−	−
N/A	1000	10000	−	−

Detection Limit (DL) = 1 ug/L
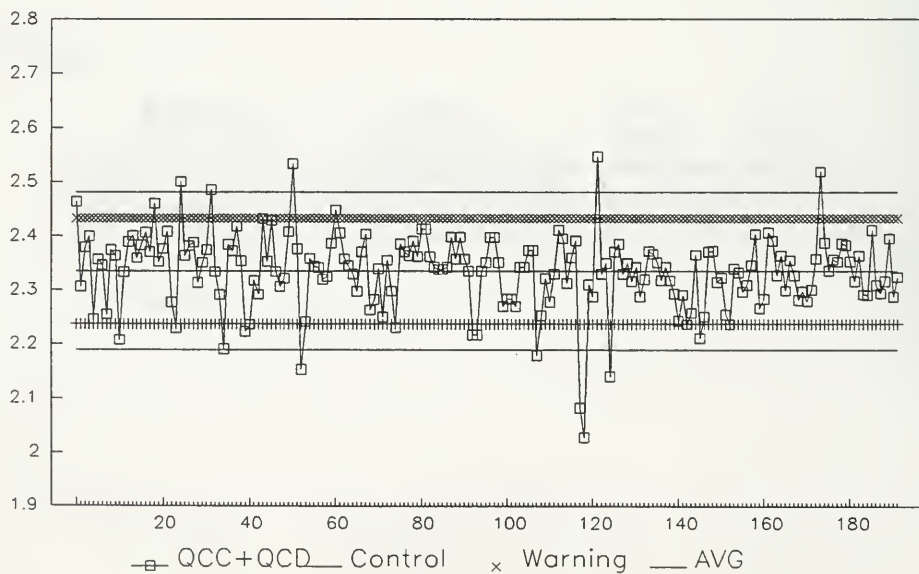
Titanium, QCC (TBMPRE)
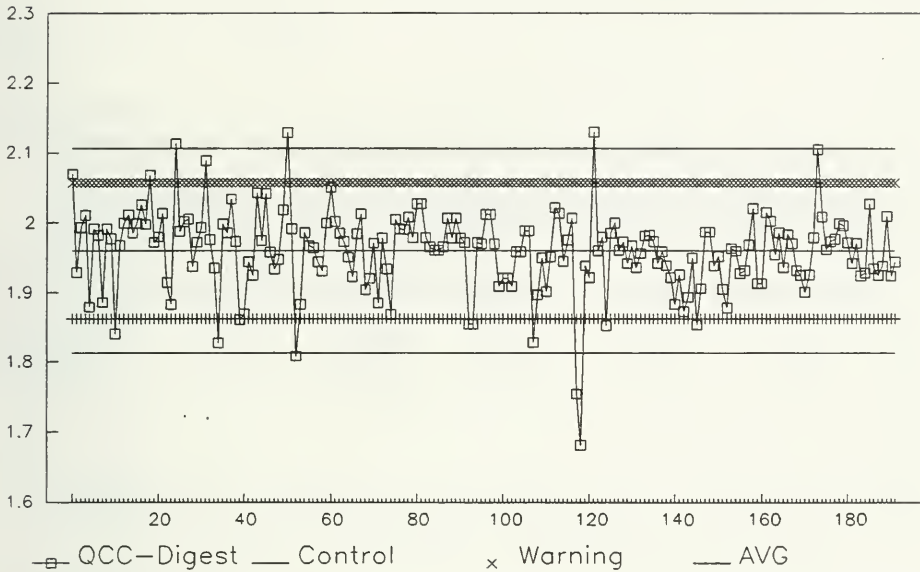
Titanium, QCD (TBMPRE)
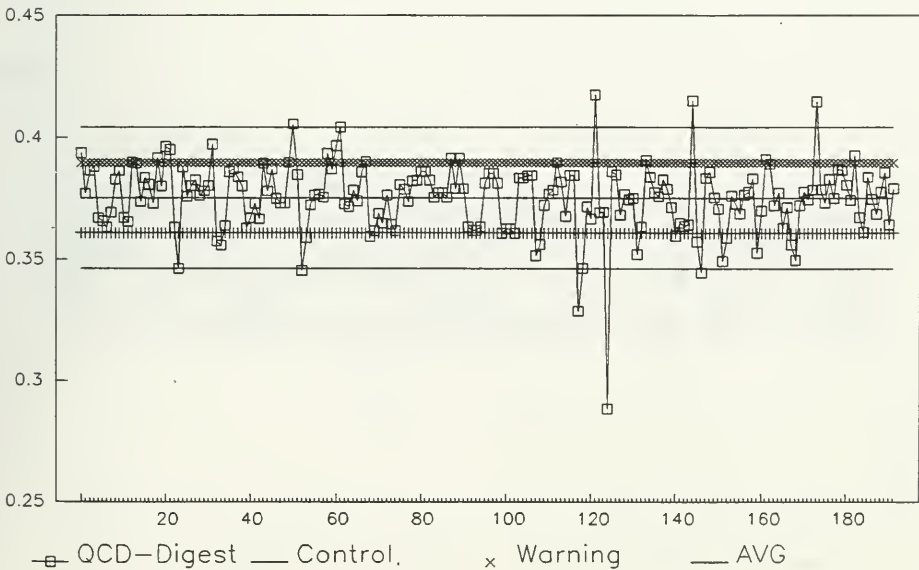
Titanium, QC Difference (TBMPRE)

Titanium, QC Sum (TBMPRE)

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TBMPRE – VANADIUM (TOTAL) – VVUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 0.5 – 25,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.972	98.6	2.2
QCD:	192	0.400	0.386	96.5	3.0
QCC+QCD:	192	2.400	2.358	98.3	2.3
QCC−QCD:	192	1.600	1.585	99.1	2.3

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.036$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
177	0.5	50	1.90	0.2500
N/A	50	500	–	–
N/A	500	5000	–	–

Detection Limit (DL) =   0.5 ug/L

Vanadium, QCC (TBMPRE)

QCC—Digest ___ Control   × Warning   ___ AVG

Vanadium, QCD (TBMPRE)

QCD—Digest ___ Control   × Warning   ___ AVG
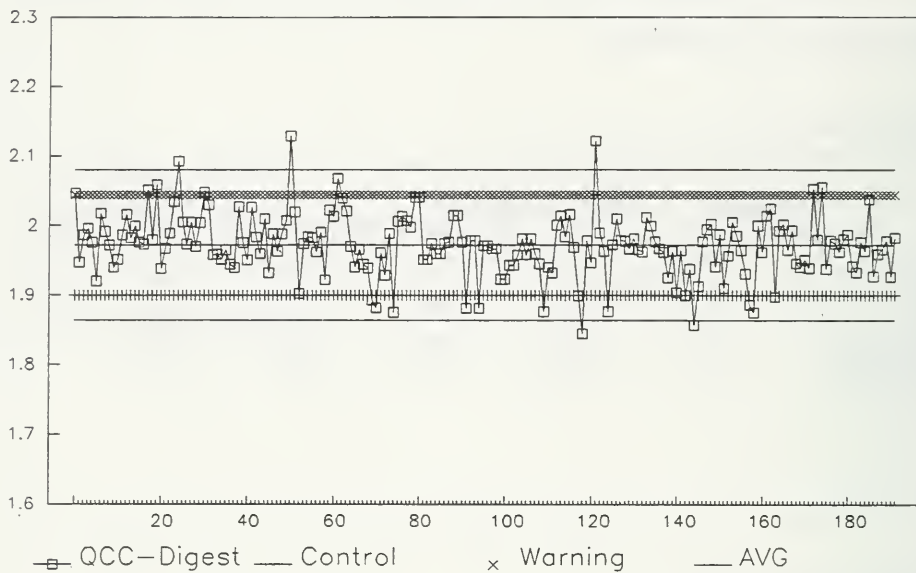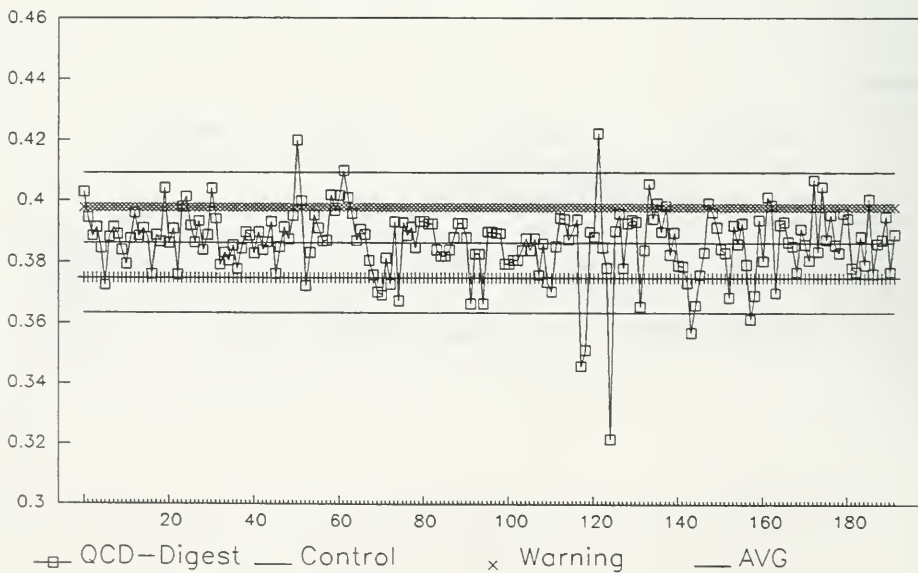
Vanadium, QC Difference (TBMPRE)

Vanadium, QC Sum (TBMPRE)
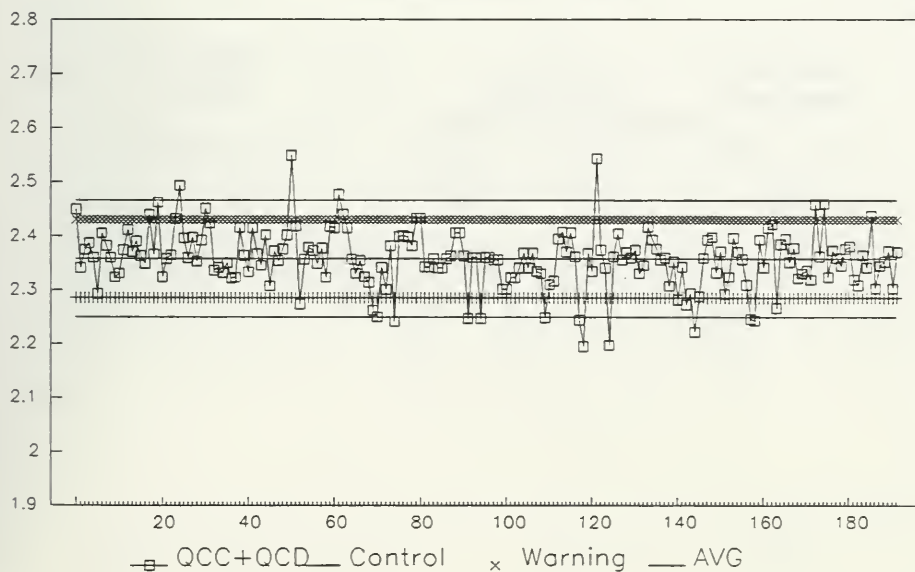
Analytical Range: 0.1 − 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	1.000	1.020	102.0	2.2
QCD:	192	0.200	0.197	98.5	2.3
QCC+QCD:	192	1.200	1.217	101.4	2.2
QCC−QCD:	192	0.800	0.823	102.9	2.3

For 1994 Control Limits:

$$Sw (C-D) = \quad 0.0187$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
175	0.1	10	0.27	0.0400
N/A	10	100	—	—
N/A	100	1000	—	—

Detection Limit (DL) = | 0.1 ug/L |

Yttrium, QCC (TBMPRE)
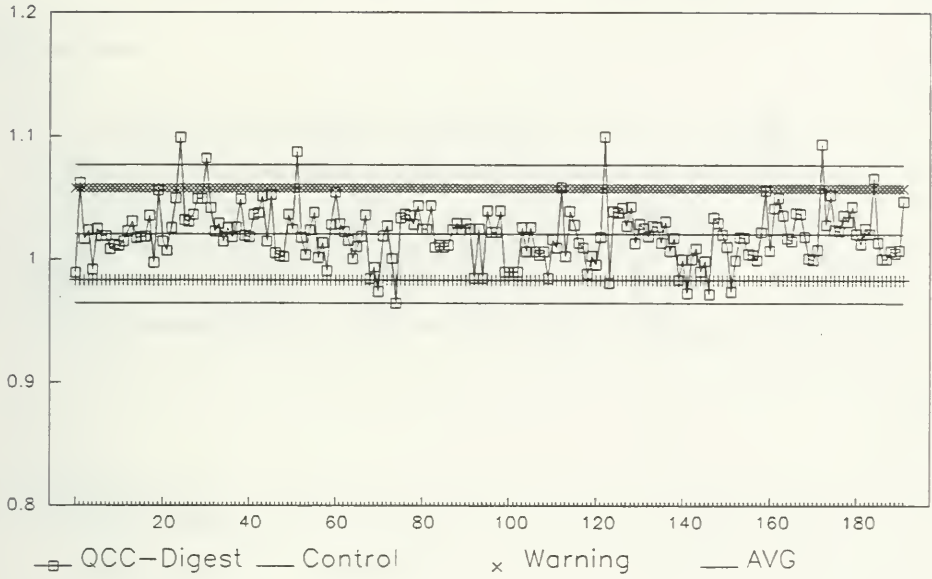
Yttrium, QCD (TBMPRE)
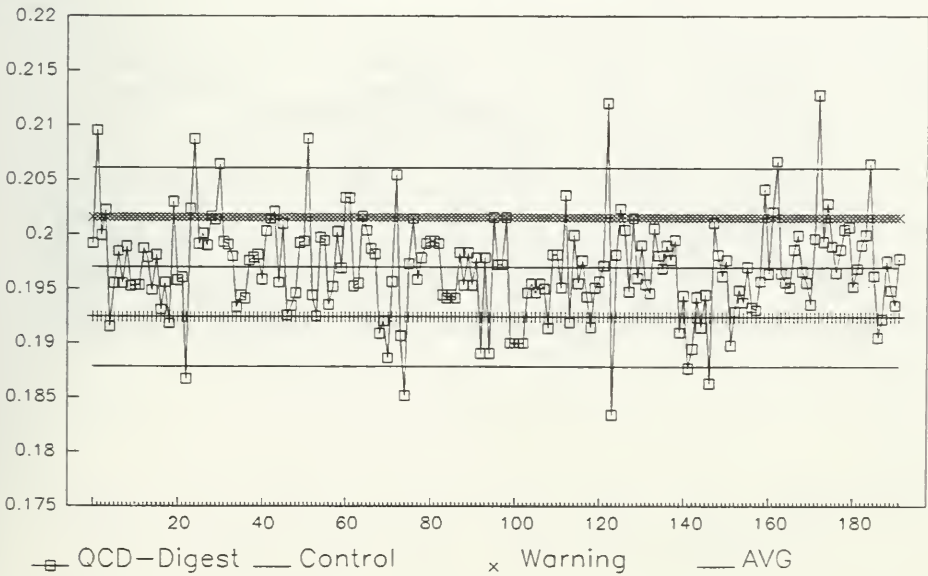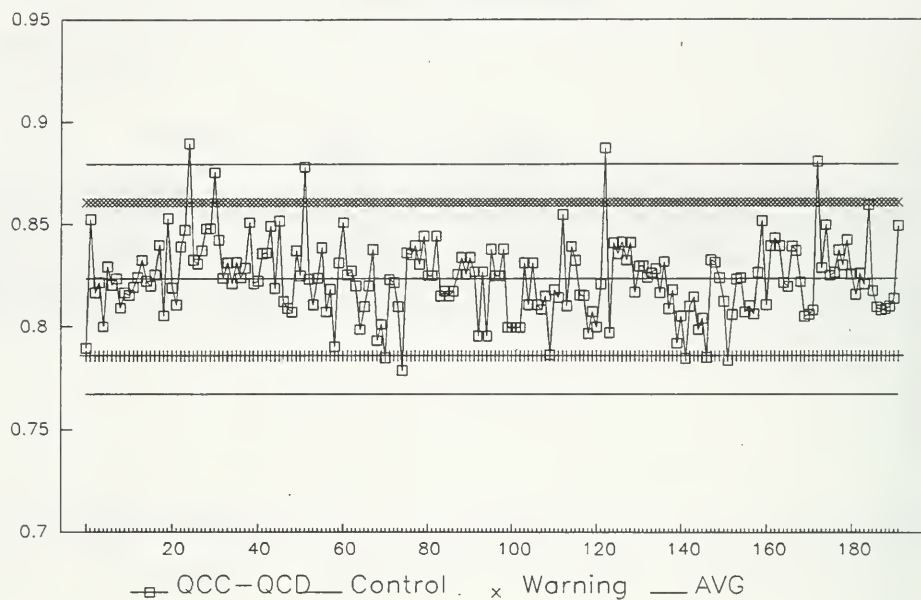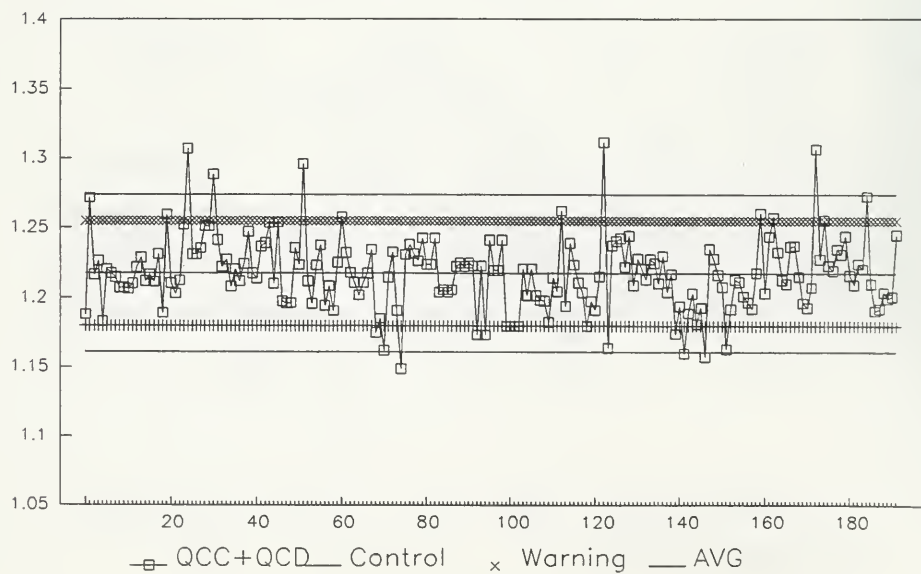
239

Yttrium, QC Difference (TBMPRE)

QCC—QCD Control x Warning AVG

Yttrium, QC Sum (TBMPRE)

QCC+QCD Control x Warning AVG

240

Analytical Range: 1 — 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	192	2.000	1.981	99.1	2.1
QCD:	192	0.400	0.394	98.5	3.8
QCC+QCD:	192	2.400	2.375	99.0	2.2
QCC−QCD:	192	1.600	1.587	99.2	2.3

For 1993 Control Limits:

$$Sw\ (C-D) = \qquad 0.036$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
146	1	100	19.00	0.5500
23	100	1000	256.0	2.60
N/A	1000	10000	—	—

Detection Limit (DL) = $\boxed{\quad 1\ ug/L \quad}$

Zinc , QCC (TBMPRE)

_□_ QCC—Digest ___ Control   × Warning   ___ AVG

Zinc, QCD (TBMPRE)

_□_ QCD—Digest ___ Control   × Warning   ___ AVG
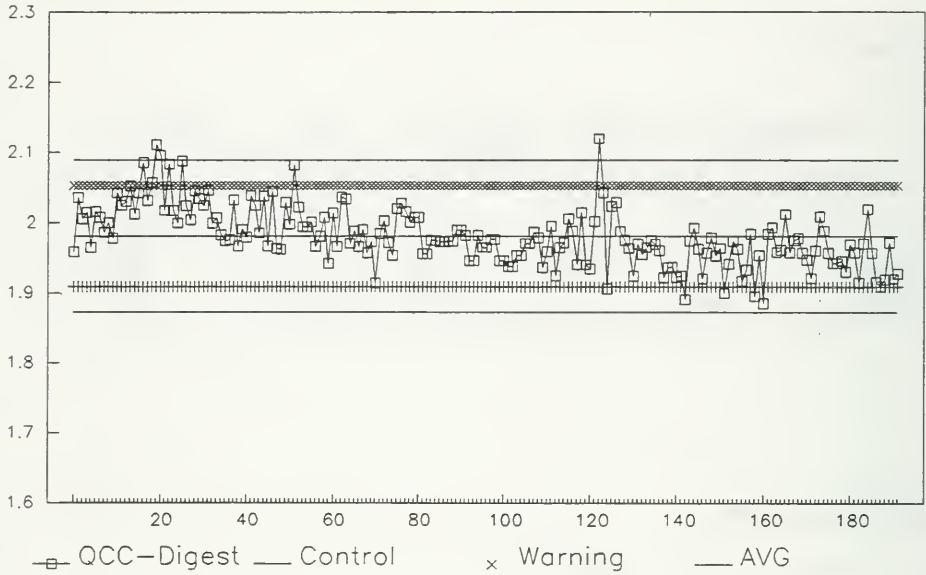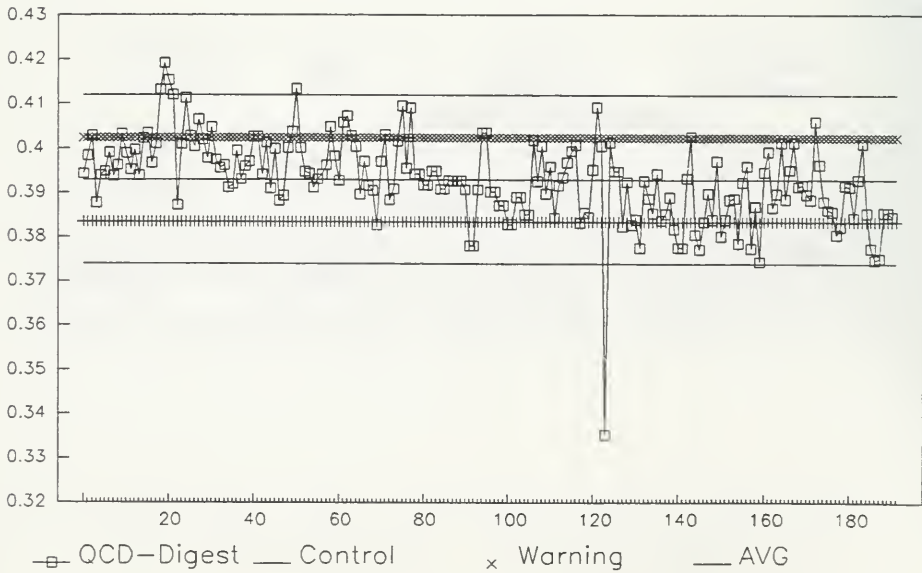
Zinc, QC Difference (TBMPRE)

Zinc, QC Difference (TBMPRE) control chart with legend: QCC−QCD, Control, Warning, AVG
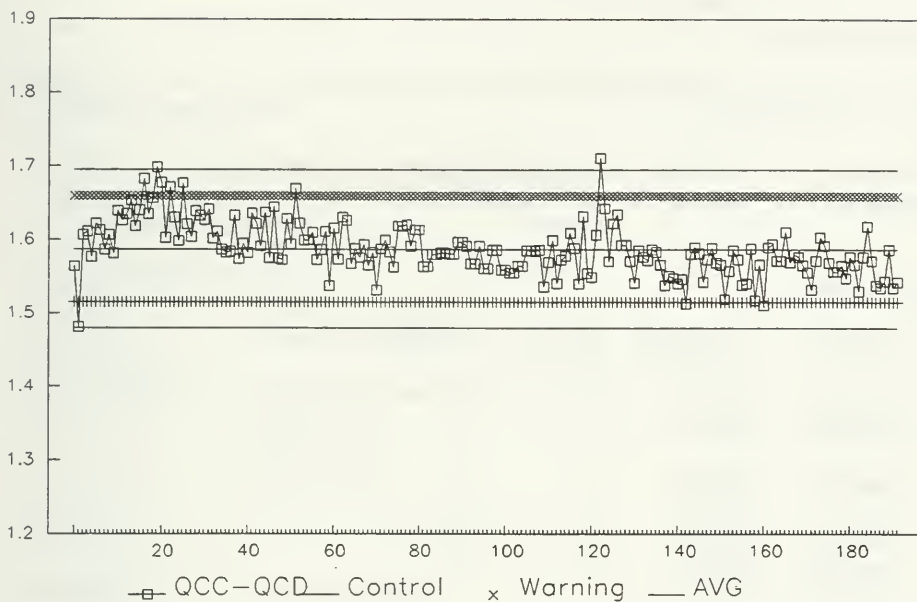
Zinc, QC Sum (TBMPRE)

Zinc, QC Sum (TBMPRE) control chart with legend: QCC+QCD, Control, Warning, AVG
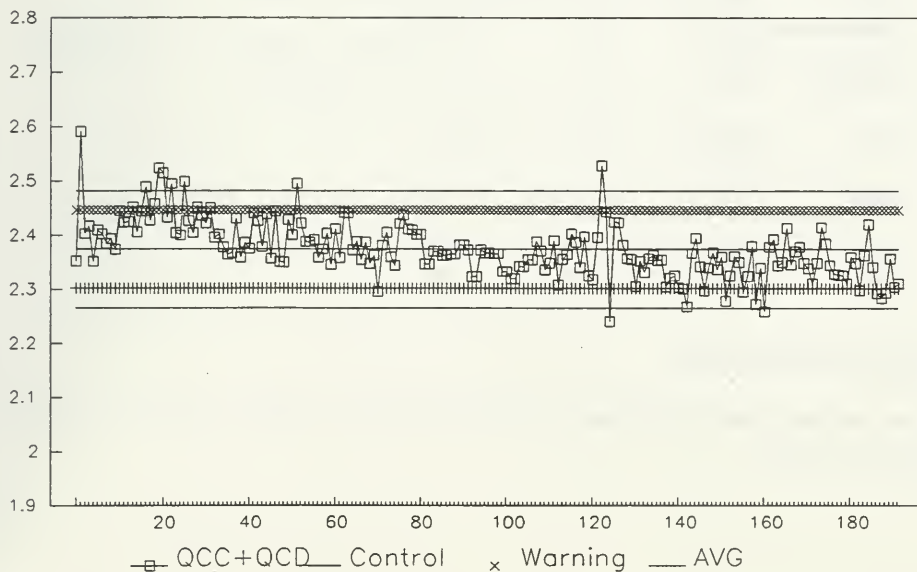
TRACE METALS WITHOUT PRECONCENTRATION

IDENTIFICATION:

Method Title: The Determination of Trace Metals in Industrial Effluents and Landfill Leachates by ICP-AES.

WorkStation:	TBMTR	Method Introduced:	May 1988
Method Code:	E6015A	Current Revision:	July 1993
		Section:	Trace Contaminants

PARAMETERS:

Element	LIS Code	W (ug/L)	T (ug/L)
Yttrium	YYUT	1	5
Beryllium	BEUT	1	5
Cadmium	CDUT	2	10
Cobalt	COUT	2	10
Manganese	MNUT	2	10
Strontium	SRUT	2	10
Vanadium	VVUT	2	10
Chromium	CRUT	5	25
Copper	CUUT	5	25
Barium	BAUT	5	25
Molybdenum	MOUT	5	25
Nickel	NIUT	5	25
Titanium	TIUT	5	25
Zinc	ZNUT	10	50
Lead	PBUT	20	100
Aluminium	ALUT	50	250
Iron	FEUT	50	250

SAMPLE TYPE/MATRIX:

Aqueous samples of industrial effluents or leachates that may or are likely to contain high levels of inorganic elements.

ANALYTICAL PROCEDURE:

Samples are subjected to nitric-hydrochloric acid digestion followed by analysis by ICP-AES.

INSTRUMENTATION:

Thermo Jarrell Ash (ICAP61) Inductively Coupled Plasma Spectrometer; Linear, two-point calibration.

QUALITY ASSURANCE:

Controls: Blank, QCC, QCD

Ref. Materials: ERA PP-42 Trace Metals Standard
                   EPA ICAP 7
                   EPA ICAP 19
                   NRC SLRS-1

Drift: QCC analyzed every 20 samples

Duplicates: 1 per 20 samples

Interlabs: MOEE Blind Audit Program (Bimonthly)
                   Great Lakes Action Program(GLAP, 2x annually)
                   CAEAL Certification Program (2x annually)

Reporting: Units: ug/L (ppb)
                   Sig. Figures: 2

TBMTR − ALUMINUM (TOTAL) − ALUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 50 − 500,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	66	10.000	10.015	100.2	7.5
QCD:	66	2.000	2.127	106.4	33.0
QCC+QCD:	66	12.000	12.142	101.2	10.2
QCC−QCD:	66	8.000	7.888	98.6	9.5

For 1994 Control Limits:

$Sw (C-D) =$ 0.748

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
60	50	5000	400.	88.
N/A	5000	50000	−	−
N/A	50000	500000	−	−

Detection Limit (DL) = 50 ug/L

Aluminum, QCC (TBMTR)

Legend: □ QCC-Digest ── Control   × Warning   ── AVG

Aluminum, QCD (TBMTR)

Legend: □ QCD-Digest ── Control   × Warning   ── AVG

Aluminum, QC Difference (TBMTR)

□ QCC−QCD ── Control   × Warning ── AVG

Aluminum, QC Sum (TBMTR)

□ QCC+QCD ── Control   × Warning ── AVG

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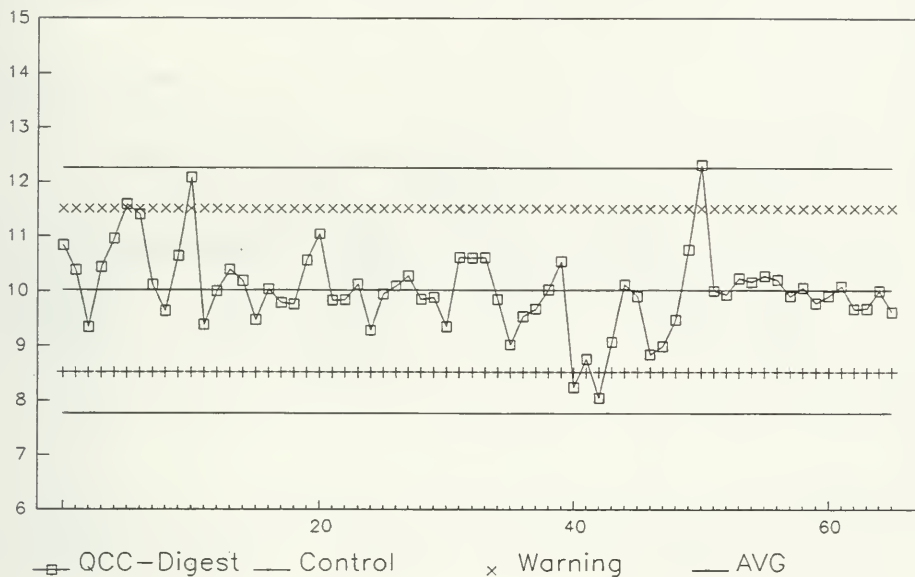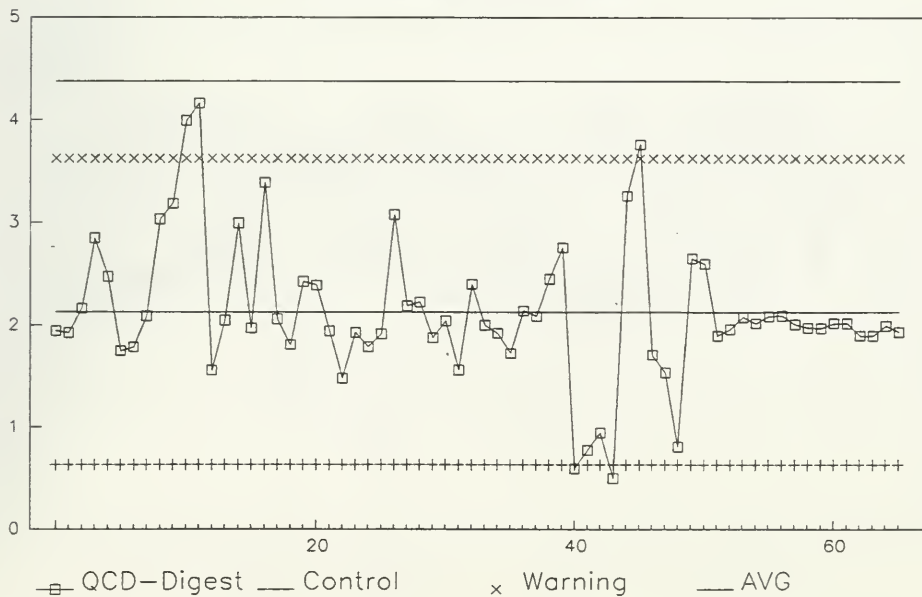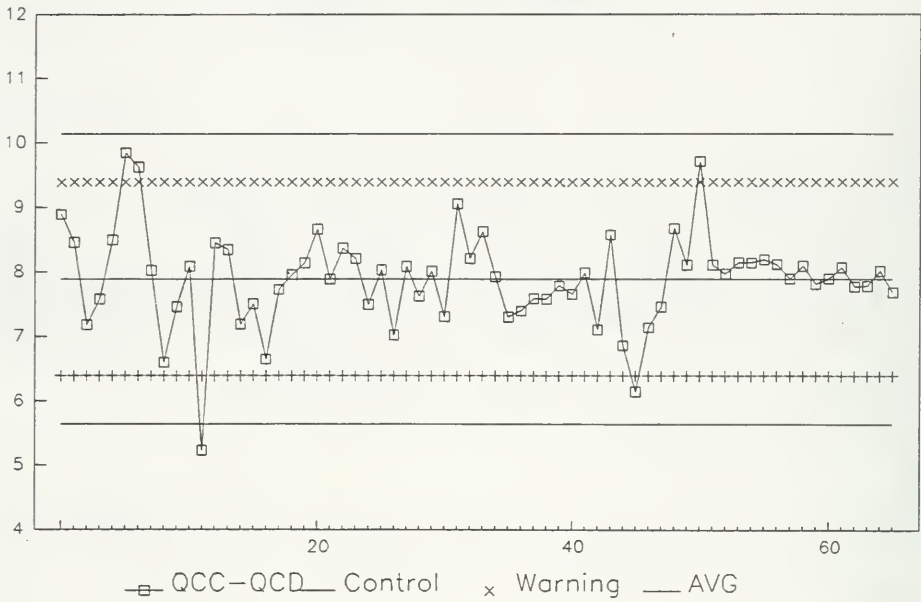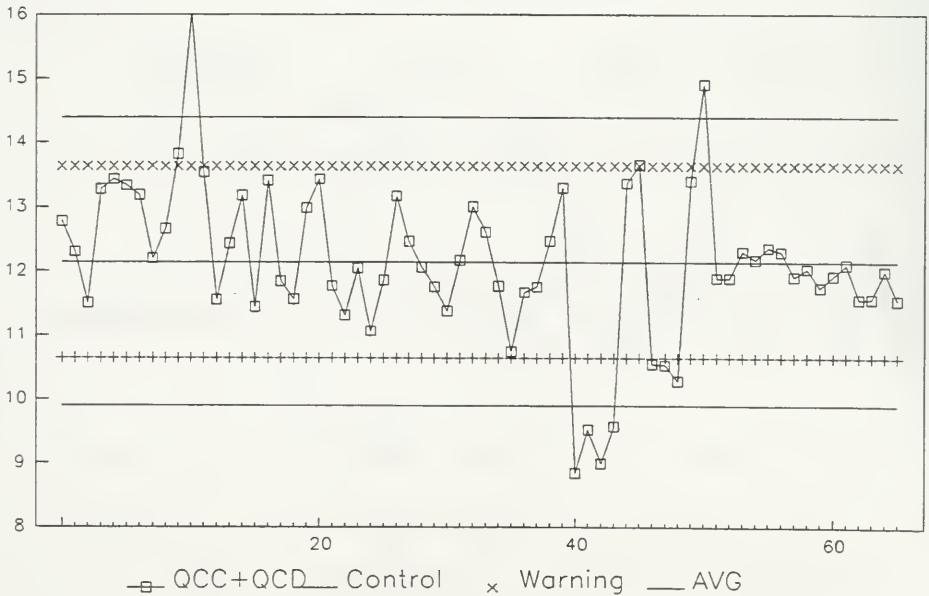
TBMTR — BARIUM (TOTAL) — BAUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range — 5–10,000 ug/L

Calibration Control:

	Number of Data	Expected Conc.	Avg. Conc. Measured	% Recovery	Standard Deviation
QCC:	65	1.000	1.001	100.1	2.9
QCD:	65	0.200	0.203	101.5	7.2
QCC+QCD:	65	1.200	1.204	100.3	3.1
QCC−QCD:	65	0.800	0.798	99.8	3.2

For 1994 Control Limits:

$$Sw\ (C-D) = \quad 0.0254$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
67	5	500	52.	3.1
N/A	500	5000	—	—
N/A	5000	10000	—	—

Detection Limit (DL) = 5 ug/L

Barium, QCC (TBMTR)
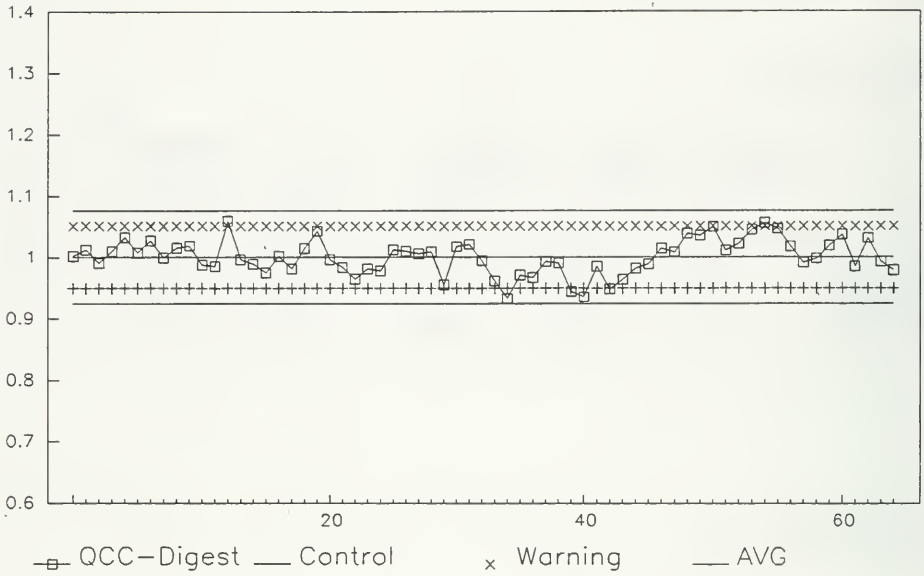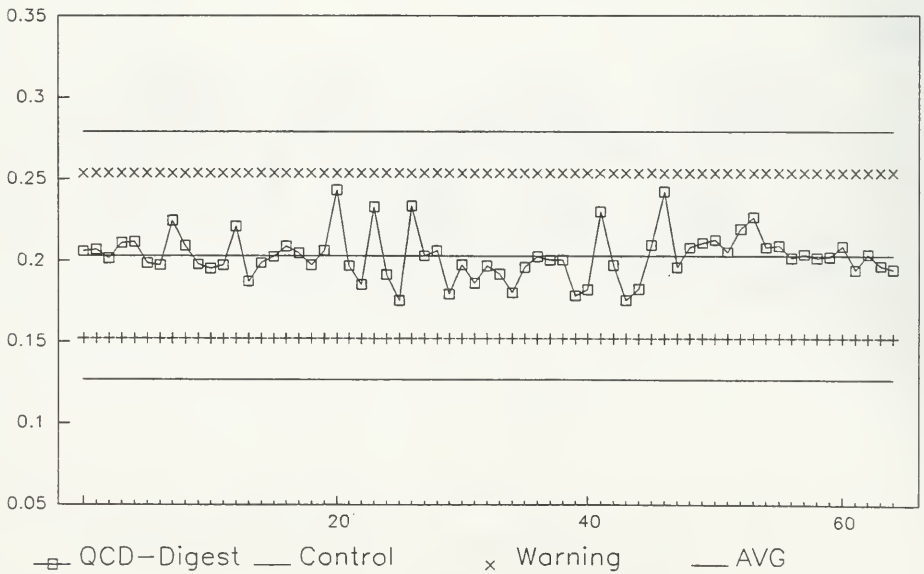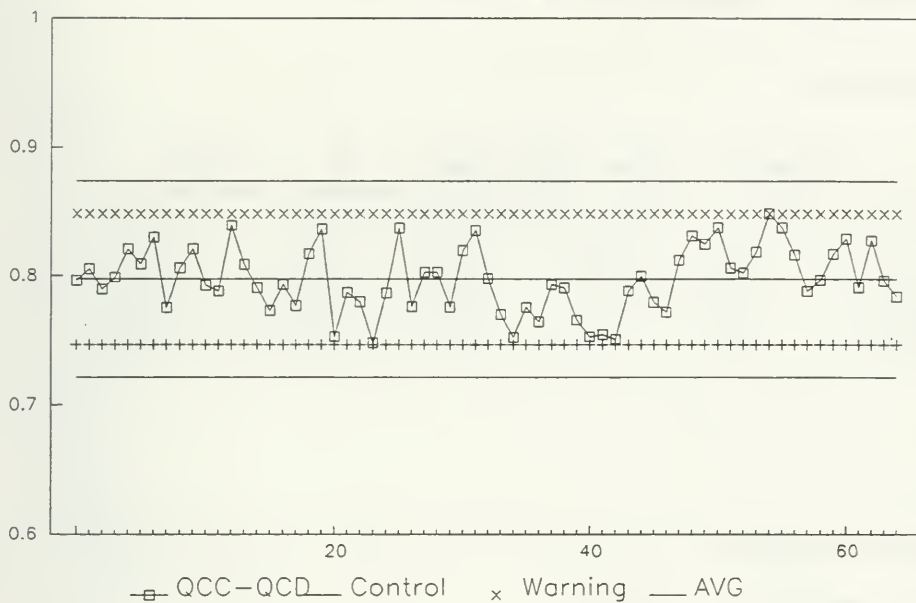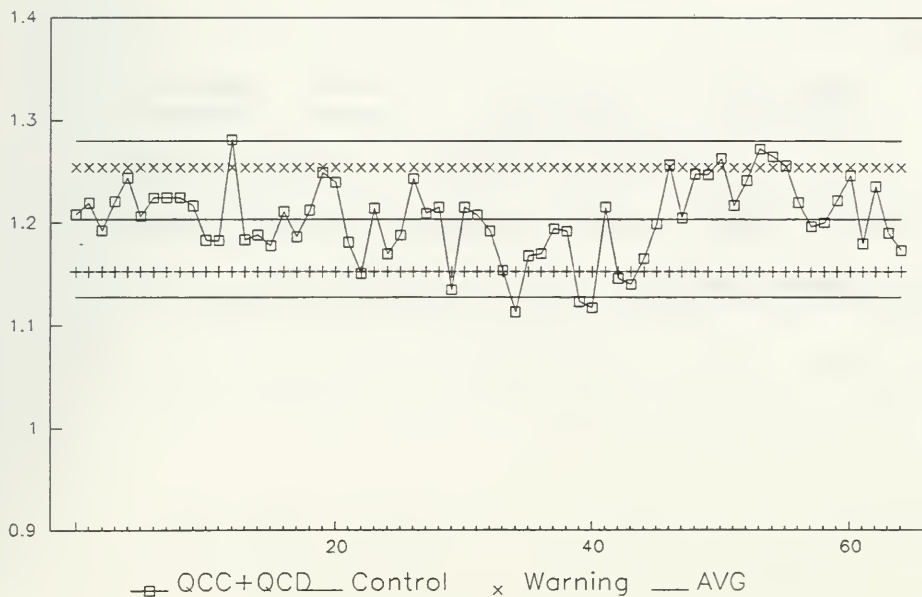
─□─ QCC−Digest ── Control   × Warning   ── AVG

Barium, QCD (TBMTR)

─□─ QCD−Digest ── Control   × Warning   ── AVG

Barium, QC Difference (TBMTR)

-ᵤ- QCC-QCD Control   × Warning __ AVG

Barium, QC Sum (TBMTR)

-ᵤ- QCC+QCD Control   × Warning __ AVG

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Analytical Range: 1 — 10,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.943	97.2	2.6
QCD:	68	0.400	0.384	96.0	2.7
QCC+QCD:	68	2.400	2.327	97.0	2.5
QCC−QCD:	68	1.600	1.559	97.4	2.7

For 1994 Control Limits:

Sw (C−D) =   0.0423

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
69	1	100	0.82	0.4200
N/A	100	1000	—	—
N/A	1000	10000	—	—

Detection Limit (DL) =   1 ug/L

Beryllium, QCC (TBMTR)

QCC–Digest ▭ Control ___ Warning × AVG ___

Beryllium, QCD (TBMTR)

QCD–Digest ▭ Control ___ Warning × AVG ___

253
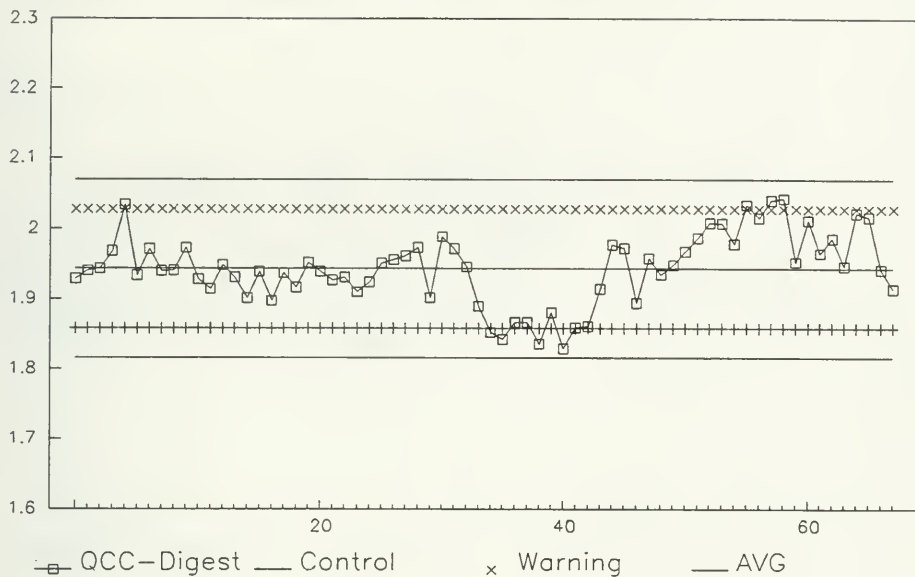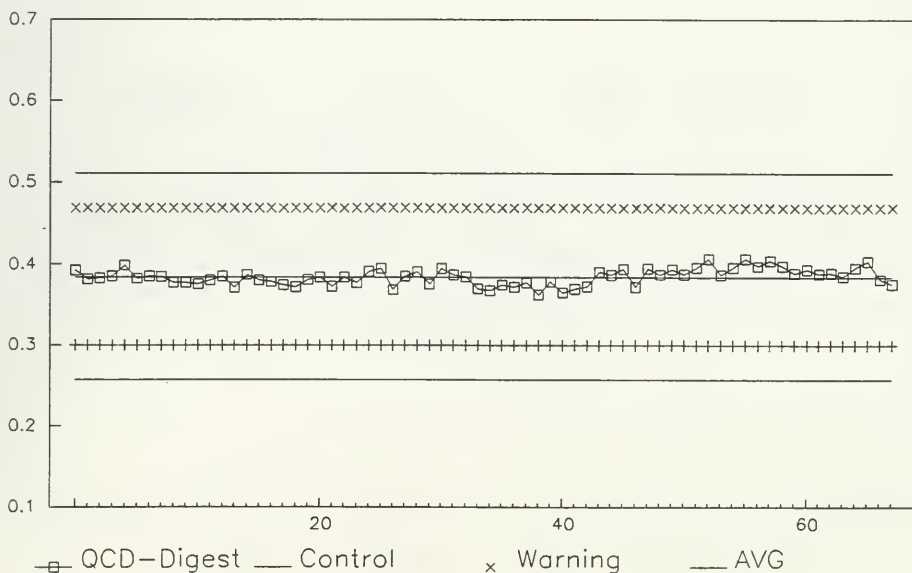
Beryllium, QC Difference (TBMTR)

Beryllium, QC Difference (TBMTR)
—□— QCC–QCD ── Control   × Warning ── AVG

Beryllium, QC Sum (TBMTR)

Beryllium, QC Sum (TBMTR)
—□— QCC+QCD ── Control   × Warning ── AVG
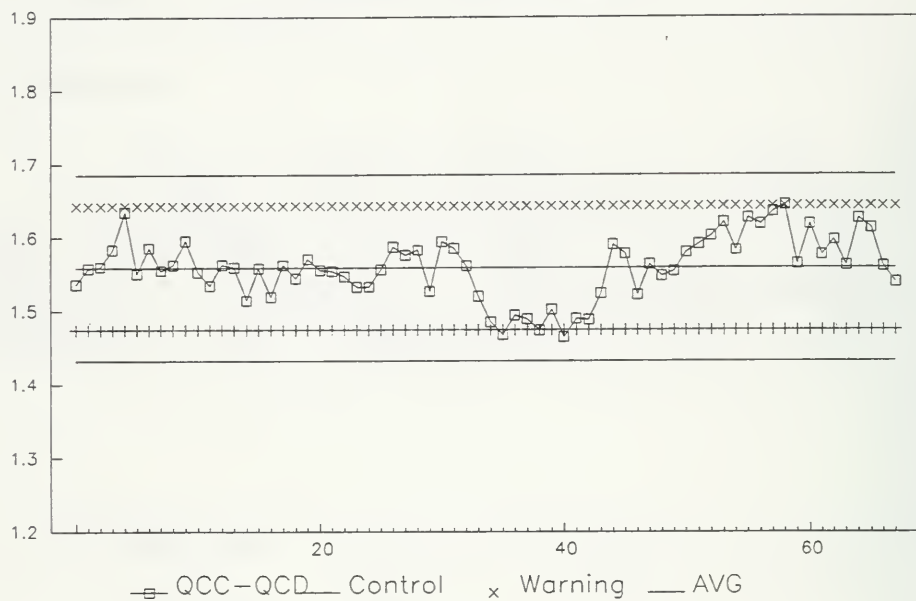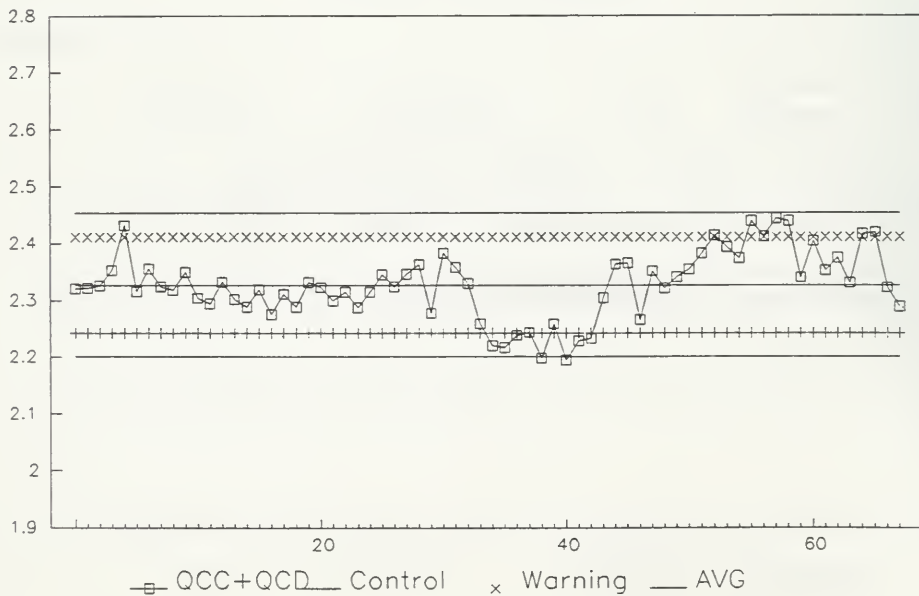
TBMTR — CADMIUM (TOTAL) — CDUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 2 — 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	1.000	0.967	96.7	2.9
QCD:	68	0.200	0.193	96.5	4.0
QCC+QCD:	68	1.200	1.160	96.7	2.9
QCC−QCD:	68	0.800	0.775	96.9	3.0

For 1994 Control Limits:

$$Sw (C-D) = 0.0235$$

Duplicates:

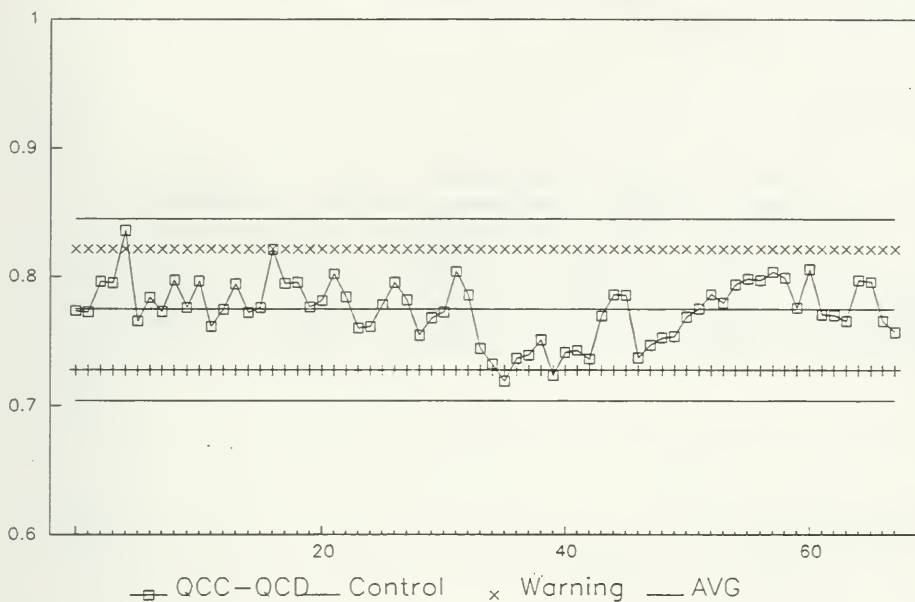| Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
66	2	200	3.4	0.79
N/A	200	2000	—	—
N/A	2000	15000	—	—

Detection Limit (DL) = 2 ug/L
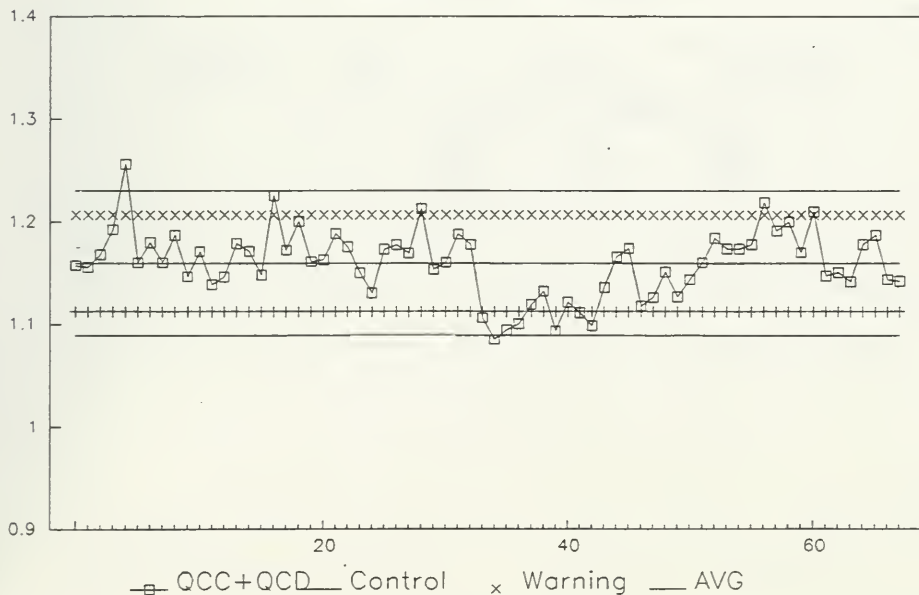
Cadmium, QCC (TBMTR)
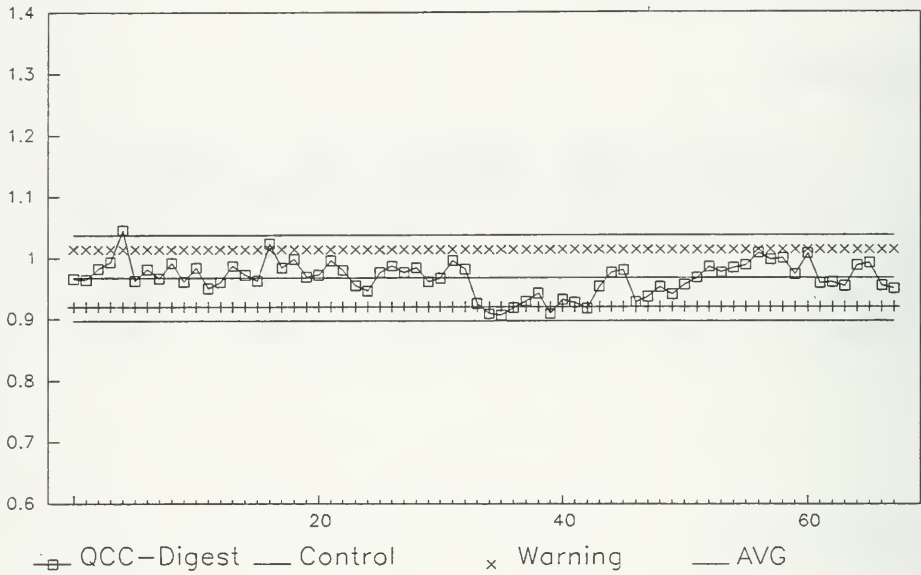
Cadmium, QCD (TBMTR)
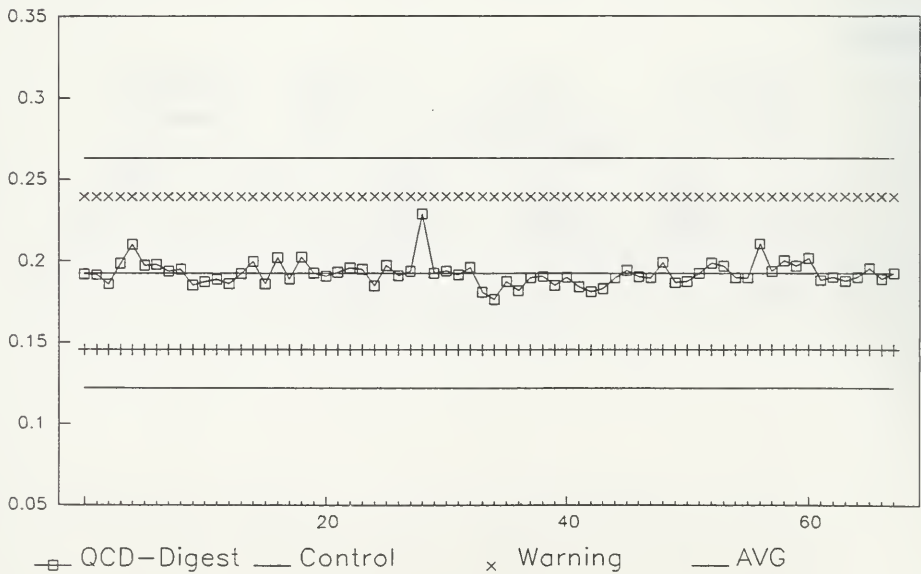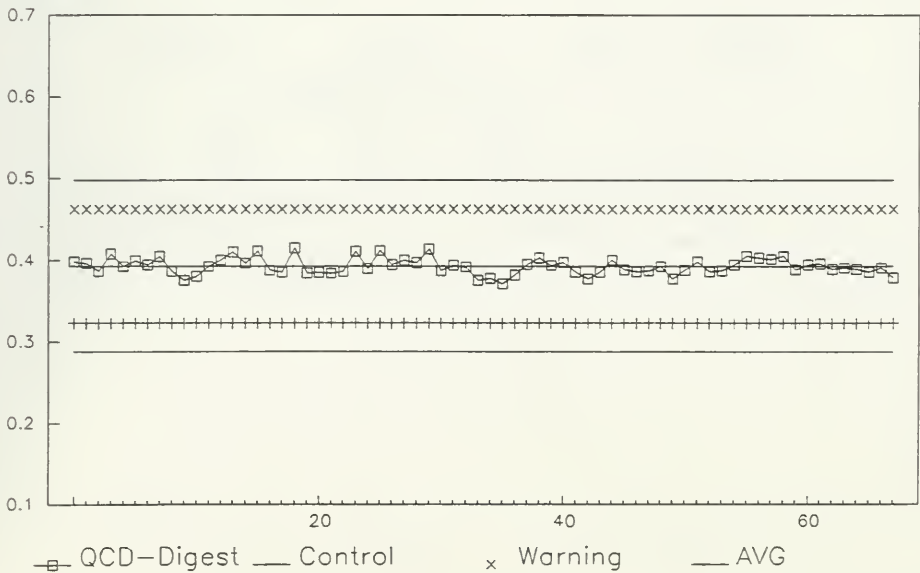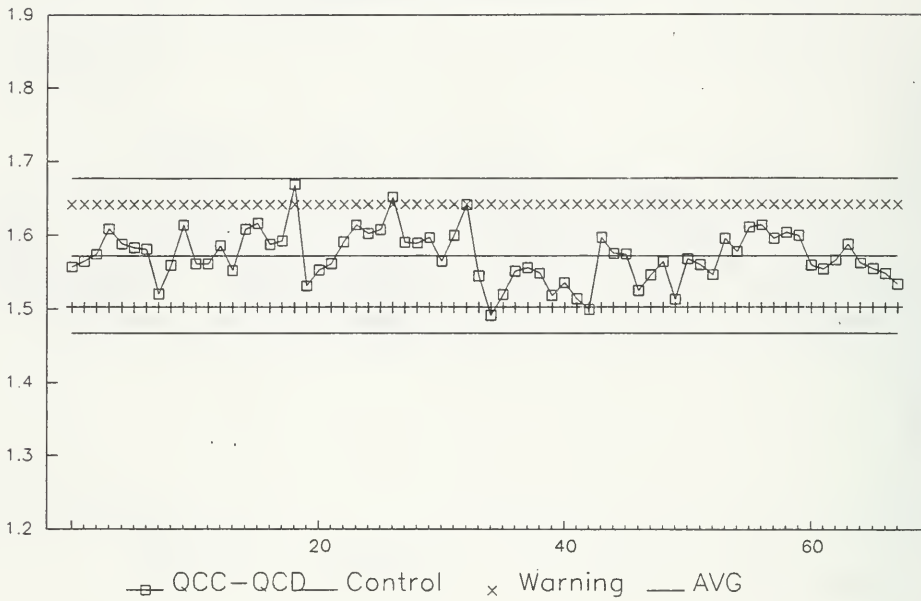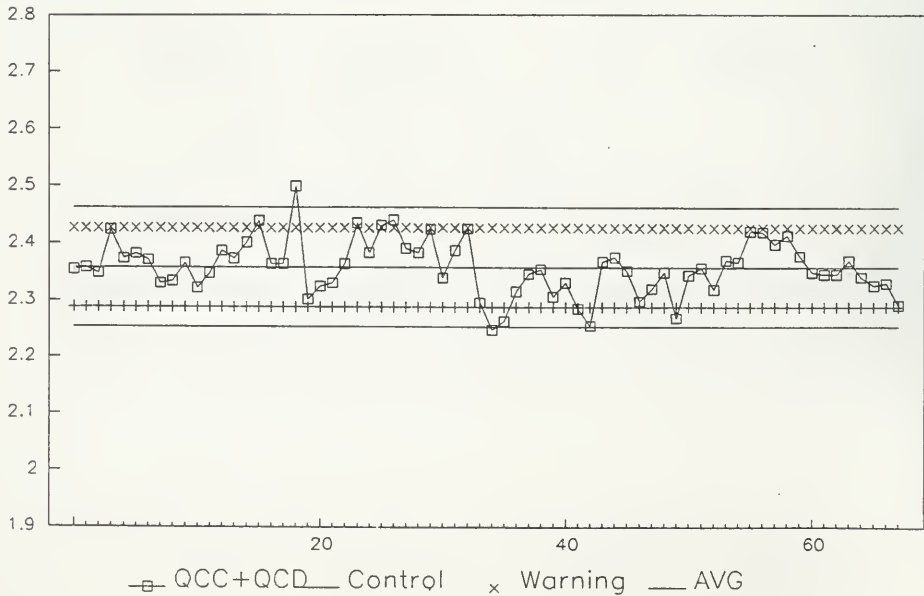
Cadmium, QC Difference (TBMTR)

Cadmium, QC Sum (TBMTR)

TBMTR – CHROMIUM (TOTAL) – CRUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 5 – 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.965	98.3	2.1
QCD:	68	0.400	0.393	98.3	2.5
QCC+QCD:	68	2.400	2.352	98.0	2.0
QCC−QCD:	68	1.600	1.572	98.3	2.2

For 1994 Control Limits:

$$Sw\ (C-D) = \quad 0.0349$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
66	5	500	9.5	1.6
N/A	500	5000	–	–
N/A	5000	15000	–	–

Detection Limit (DL) = 5 ug/L

Chromium, QCC (TBMTR)
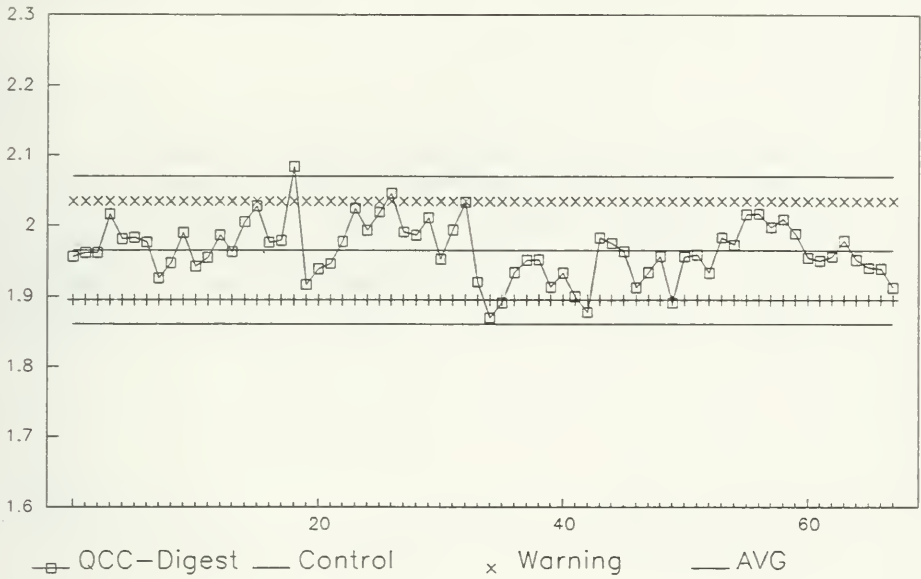
Chromium, QCD (TBMTR)

Chromium, QC Difference (TBMTR)

QCC—QCD Control × Warning AVG

Chromium, QC Sum (TBMTR)

QCC+QCD Control × Warning AVG

TBMTR − COBALT (TOTAL) − COUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 2 − 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.951	97.6	2.8
QCD:	68	0.400	0.388	97.0	3.0
QCC+QCD:	68	2.400	2.339	97.5	2.7
QCC−QCD:	68	1.600	1.563	97.7	2.9

For 1994 Control Limits:

$$Sw (C-D) = 0.0452$$

Duplicates:

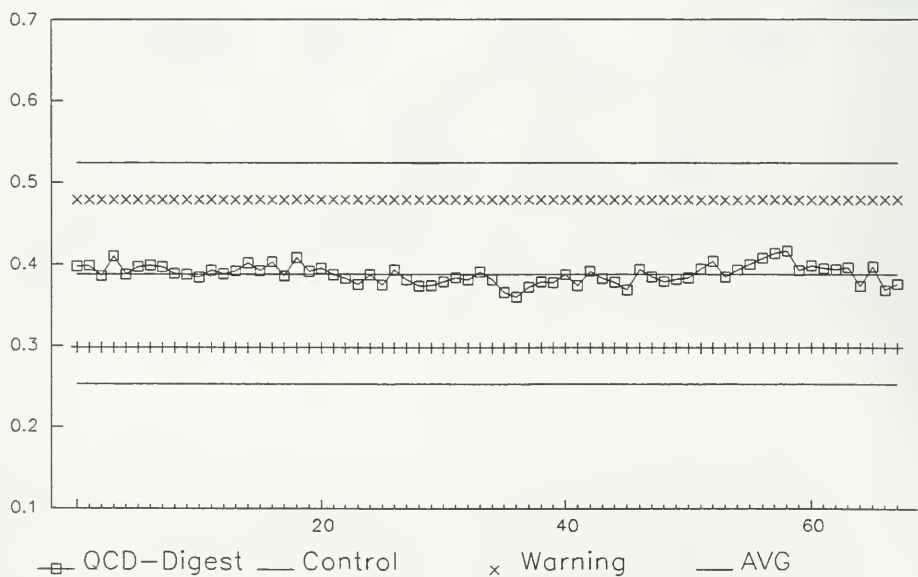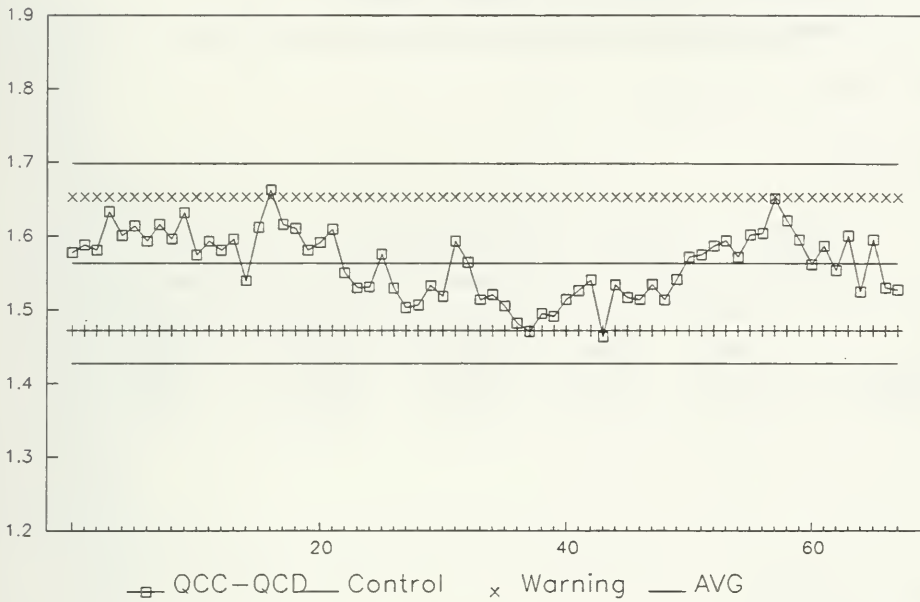| Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
67	2	200	15.	1.4
N/A	200	2000	−	−
N/A	2000	15000	−	−

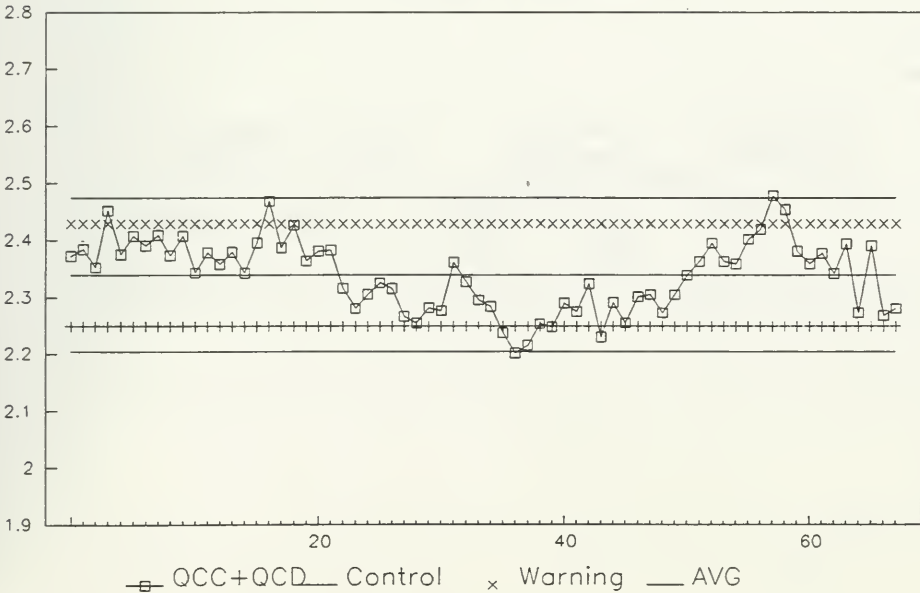Detection Limit (DL) = 2 ug/L

Cobalt, QCC (TBMTR)
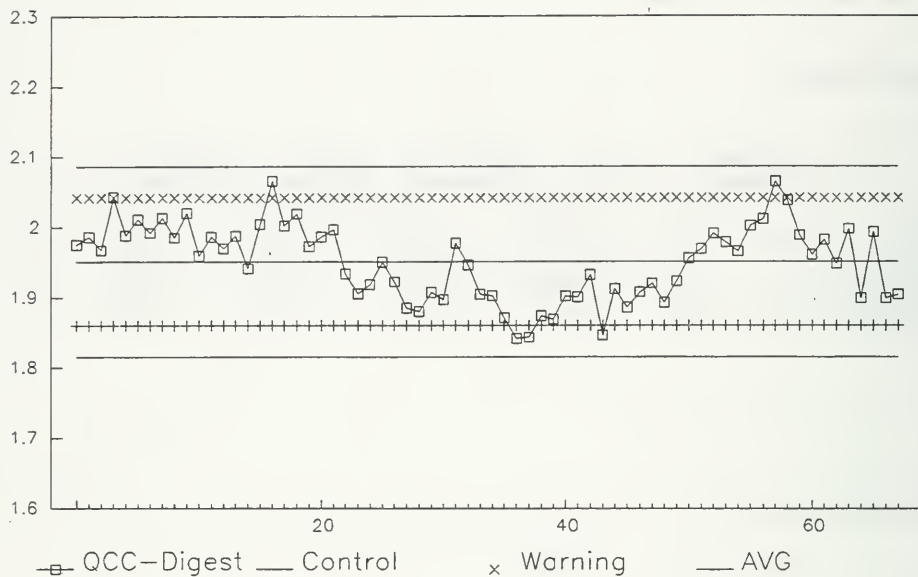
Cobalt, QCD (TBMTR)

Cobalt, QC Difference (TBMTR)

Cobalt, QC Sum (TBMTR)

TBMTR – COPPER (TOTAL) – CUUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 5 – 15,000 ug/L

Control Samples:

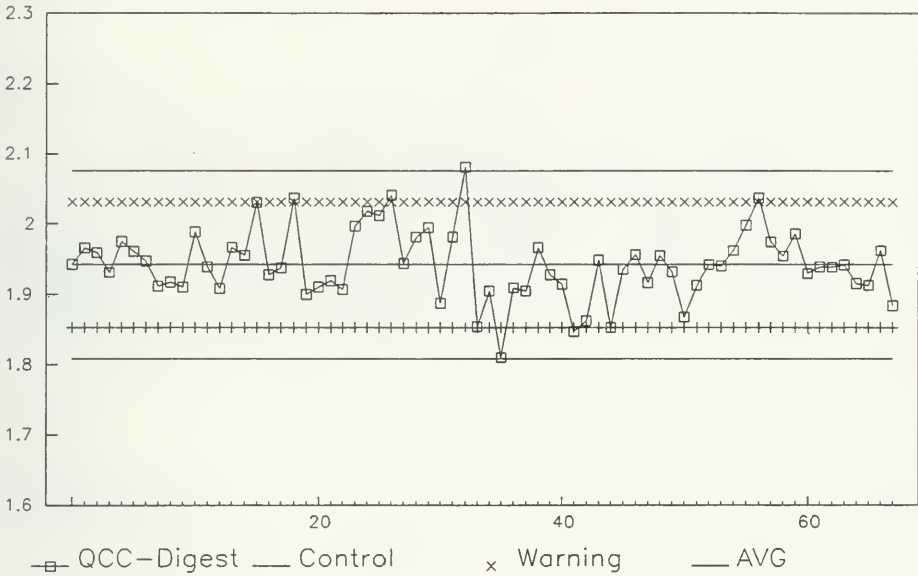|	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.942	97.1	2.6
QCD:	68	0.400	0.388	97.0	3.8
QCC+QCD:	68	2.400	2.331	97.1	2.5
QCC−QCD:	68	1.600	1.554	97.1	2.9

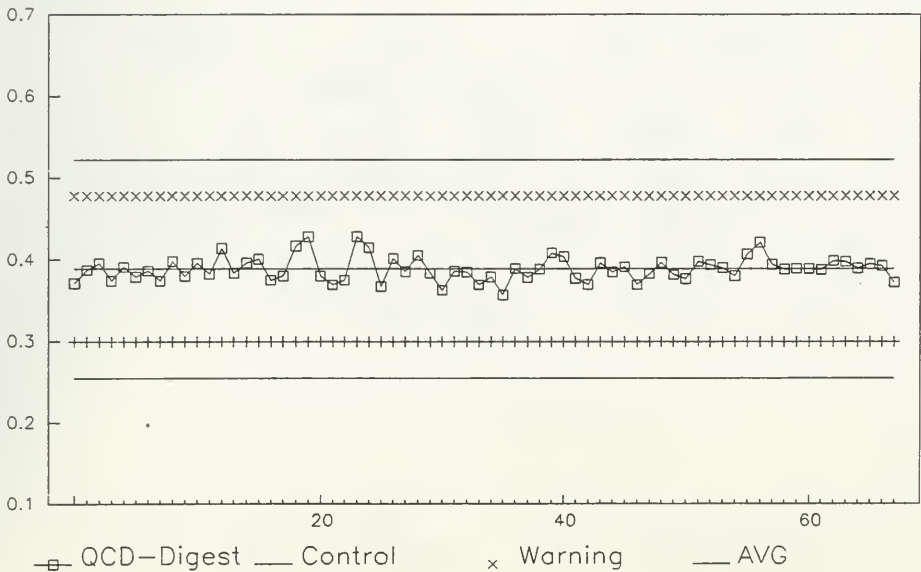For 1994 Control Limits:

$$Sw\,(C-D) = \qquad 0.0445$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
63	5	500	23.	3.2
N/A	500	5000	–	–
N/A	5000	15000	–	–

Detection Limit (DL) = | 5 ug/L |

Copper, QCC (TBMTR)

Copper, QCD (TBMTR)

Copper, QC Difference (TBMTR)

QCC-QCD — Control × Warning — AVG

Copper, QC Sum (TBMTR)

QCC+QCD — Control × Warning — AVG
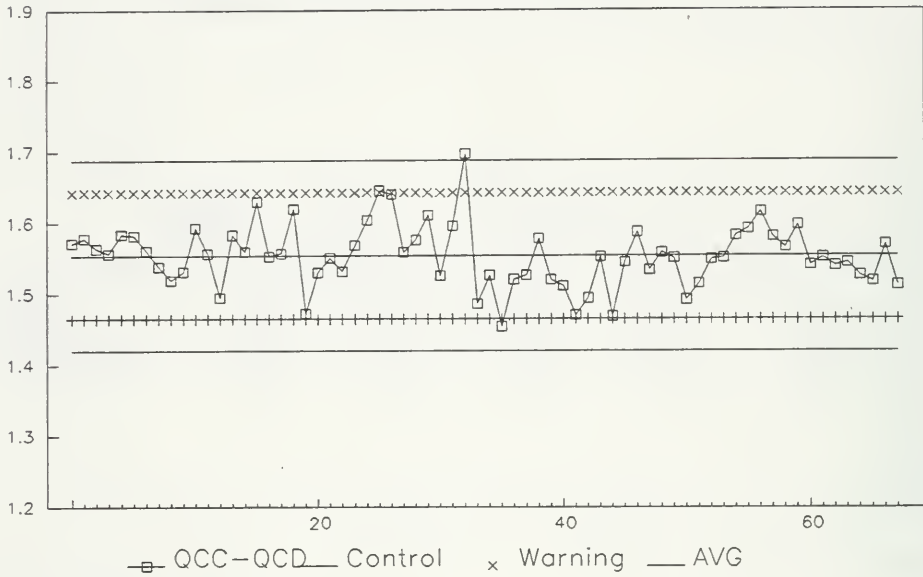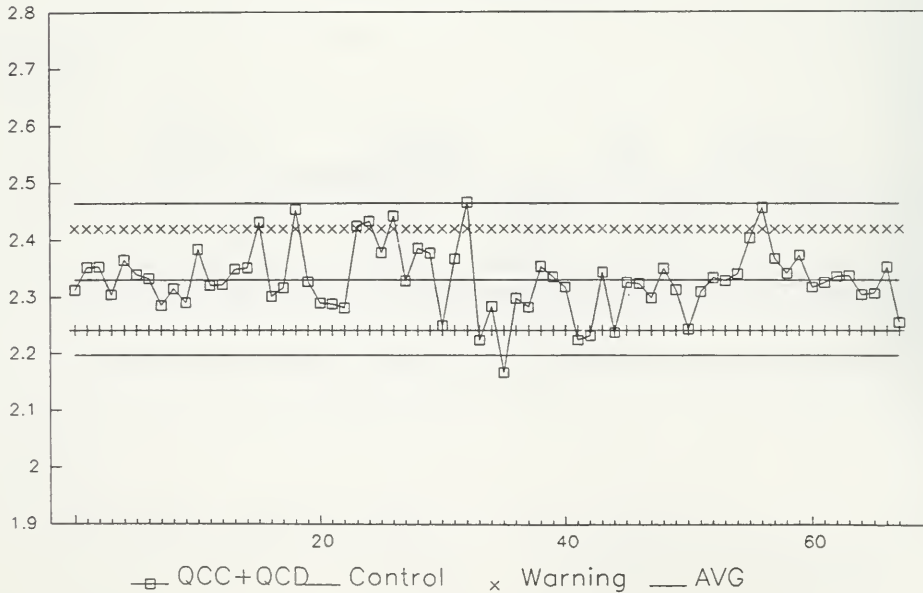
TBMTR — IRON (TOTAL) — FEUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 50 — 150,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	10.000	9.969	99.7	2.6
QCD:	68	2.000	1.992	99.6	3.7
QCC+QCD:	68	12.000	11.962	99.7	2.6
QCC−QCD:	68	8.000	7.977	99.7	2.7

For 1994 Control Limits:

$$Sw\,(C-D) = \qquad 0.218$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
54	50	5000	910	31.
9	5000	50000	25000	260.
N/A	50000	150000	—	—

Detection Limit (DL) = 50 ug/L

Iron, QCC (TBMTR)

QCC–Digest ___ Control   × Warning   ___ AVG

Iron, QCD (TBMTR)

QCD–Digest ___ Control   × Warning   ___ AVG

Iron, QC Difference (TBMTR)

QCC–QCD — Control ⨯ Warning — AVG

Iron, QC Sum (TBMTR)

QCC+QCD — Control ⨯ Warning — AVG
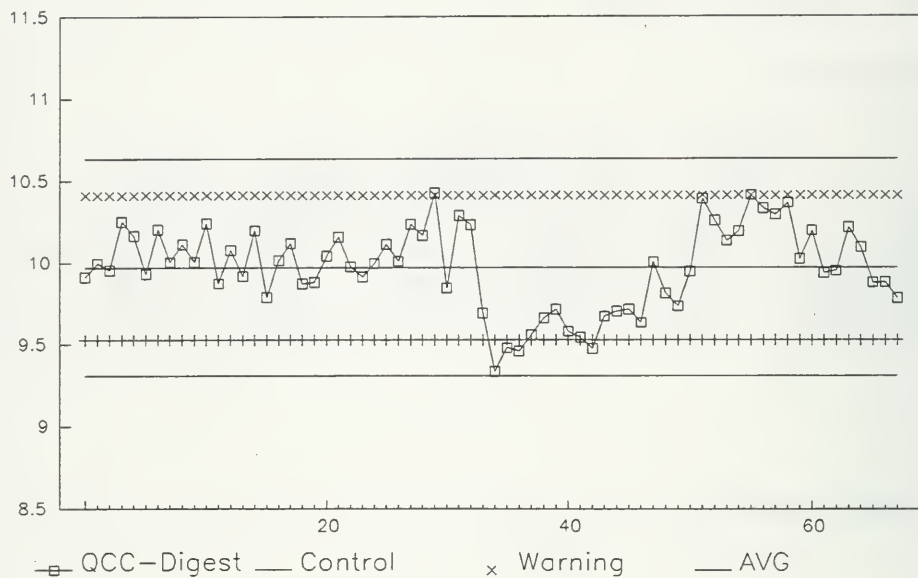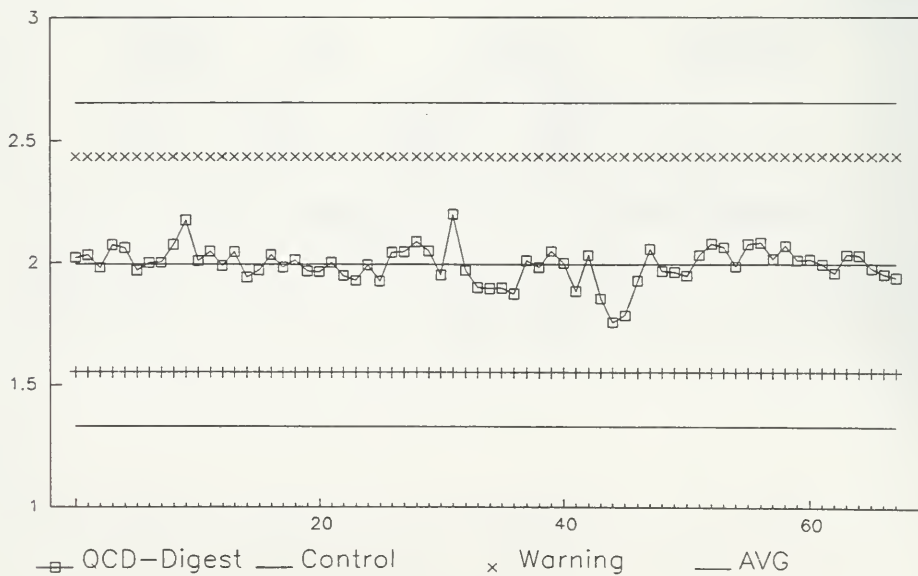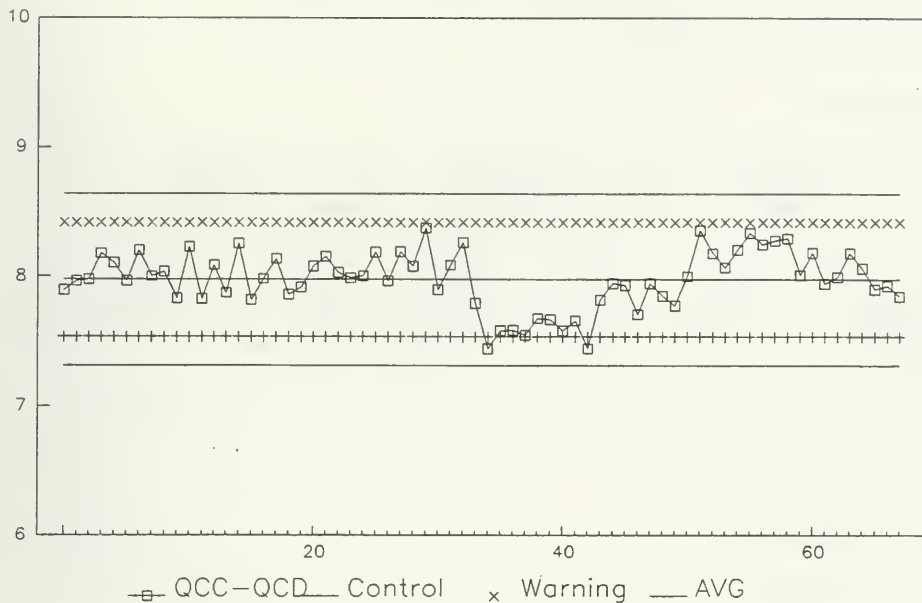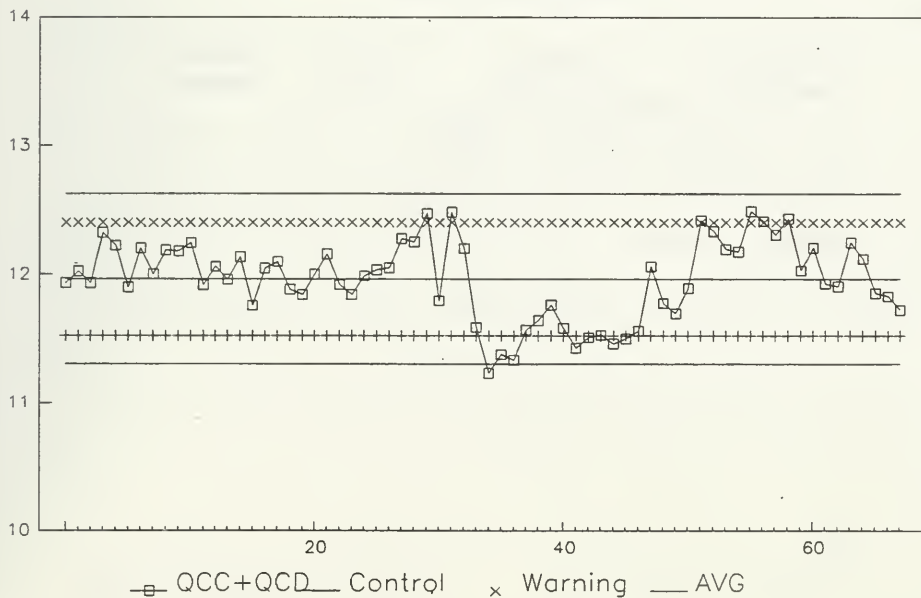
TBMTR — LEAD (TOTAL) — PBUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 20 — 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.923	96.2	3.3
QCD:	68	0.400	0.383	95.8	8.9
QCC+QCD:	68	2.400	2.306	96.1	3.6
QCC−QCD:	68	1.600	1.539	96.2	3.8

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0583$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
68	20	2000	7.0	9.5
N/A	2000	20000	—	—

Detection Limit (DL) = 20 ug/L

Lead, QCC (TBMTR)

- QCC-Digest ── Control × Warning ── AVG

Lead, QCD (TBMTR)

- QCD-Digest ── Control × Warning ── AVG

Lead, QC Difference (TBMTR)

- QCC–QCD — Control × Warning — AVG

Lead, QC Sum (TBMTR)

- QCC+QCD — Control × Warning — AVG
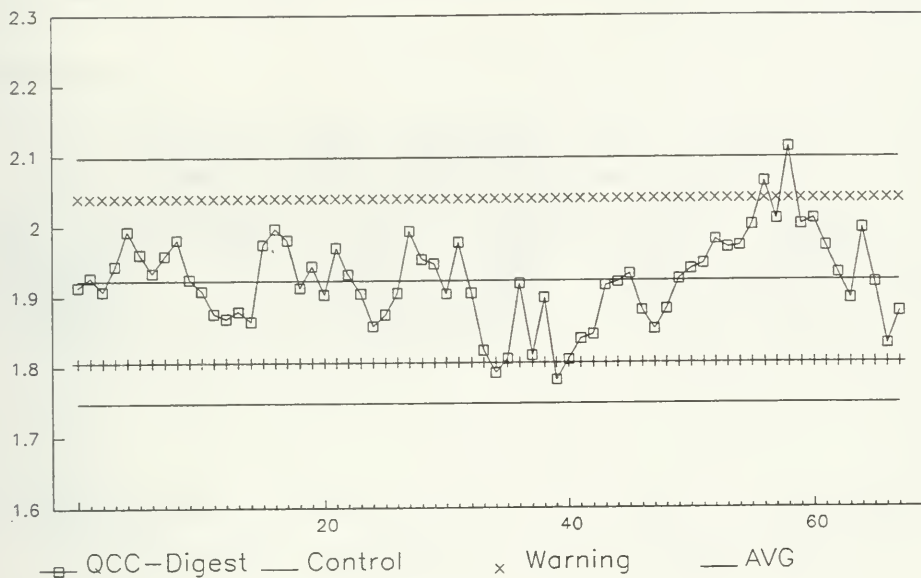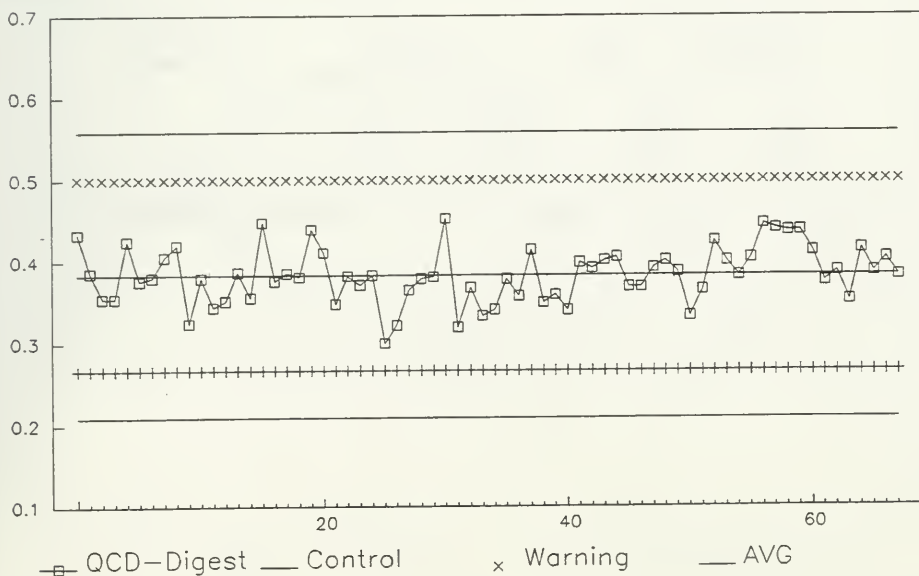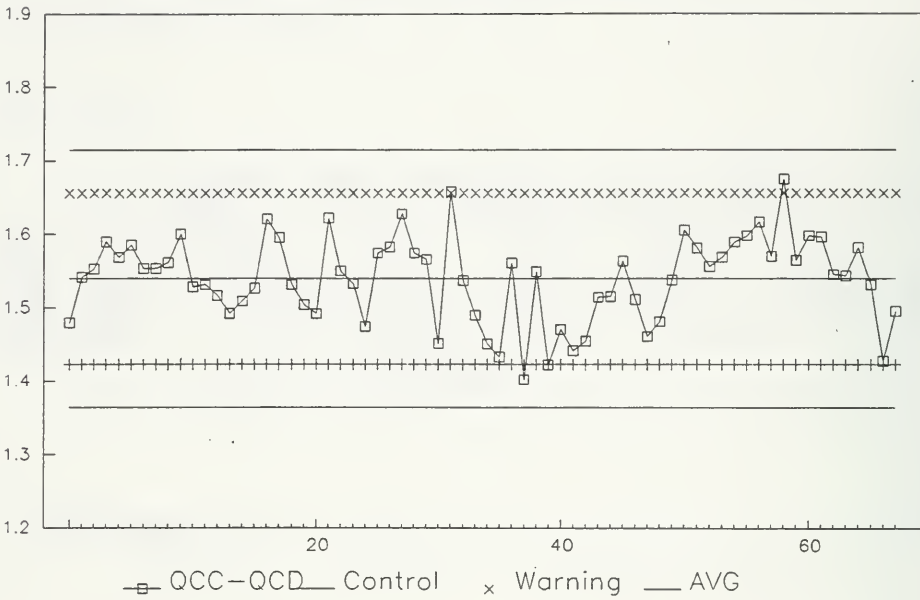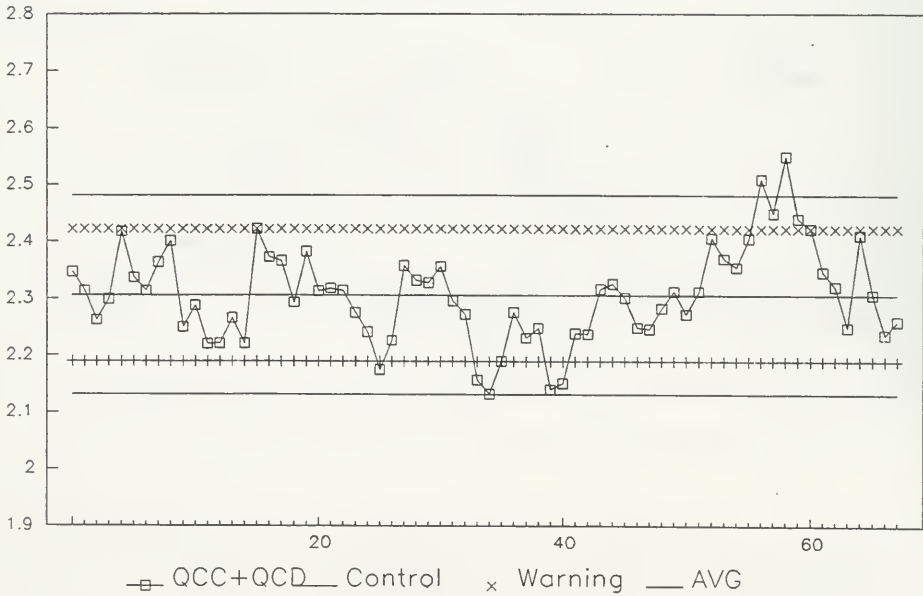
TBMTR — MANGANESE (TOTAL) — MNUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 2 – 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	67	2.000	1.946	97.3	2.6
QCD:	67	0.400	0.385	96.3	2.3
QCC+QCD:	67	2.400	2.331	97.1	2.5
QCC−QCD:	67	1.600	1.561	97.6	2.8

For 1994 Control Limits:

$$Sw\ (C-D) = \qquad 0.0439$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
47	2	200	55	1.4
13	200	2000	730	6.7
8	2000	15000	7600	110.

Detection Limit (DL) = 2 ug/L

Manganese, QCC (TBMTR)
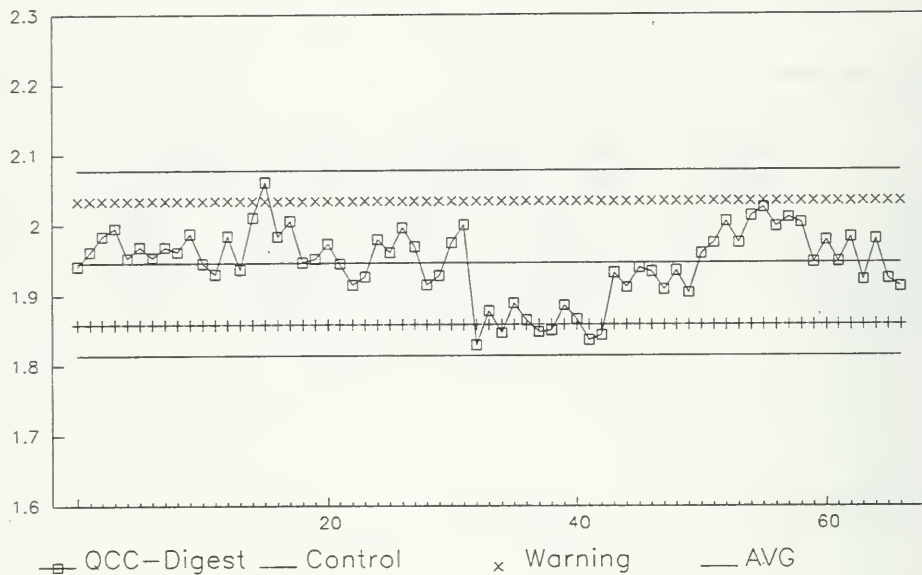
Manganese, QCD (TBMTR)

Manganese, QC Difference (TBMTR)

QCC−QCD Control × Warning ─ AVG

Manganese, QC Sum (TBMTR)

QCC+QCD Control × Warning ─ AVG
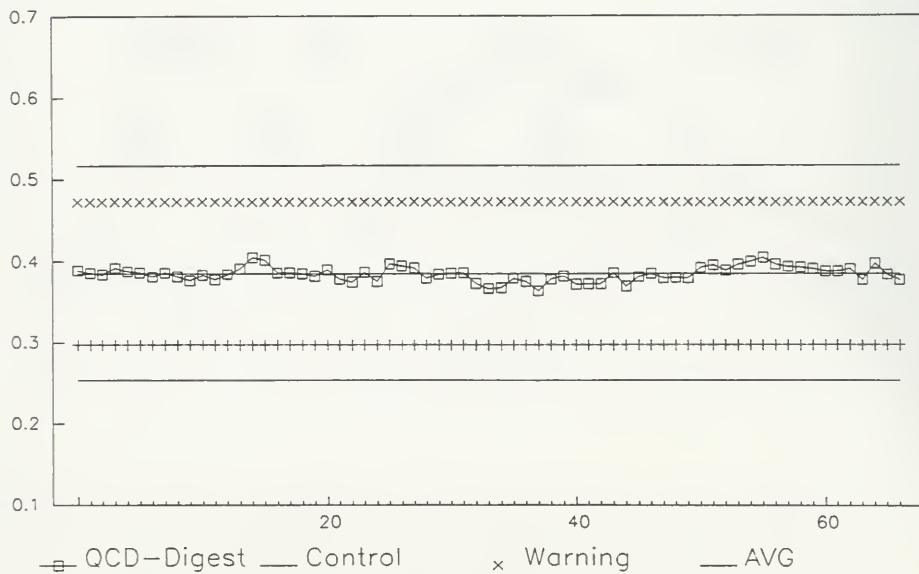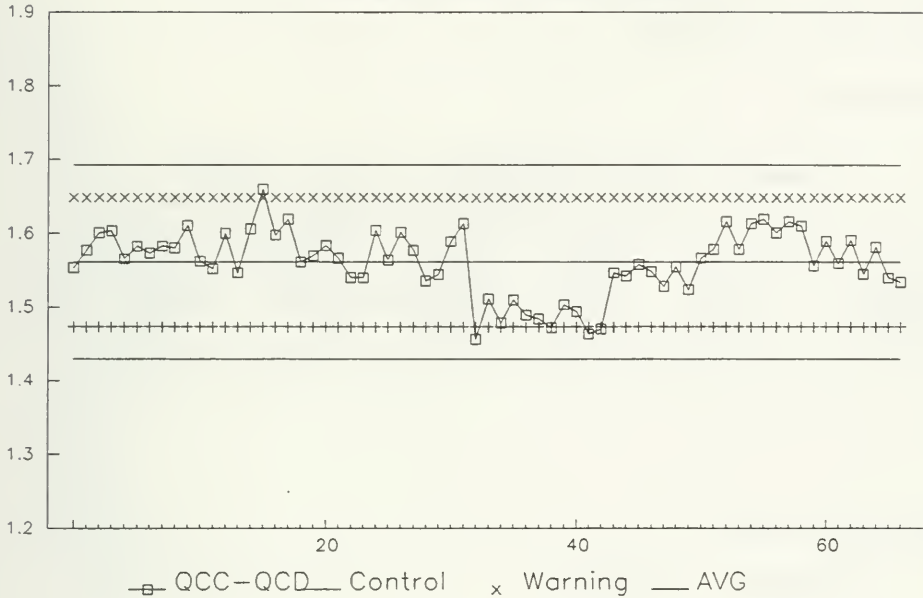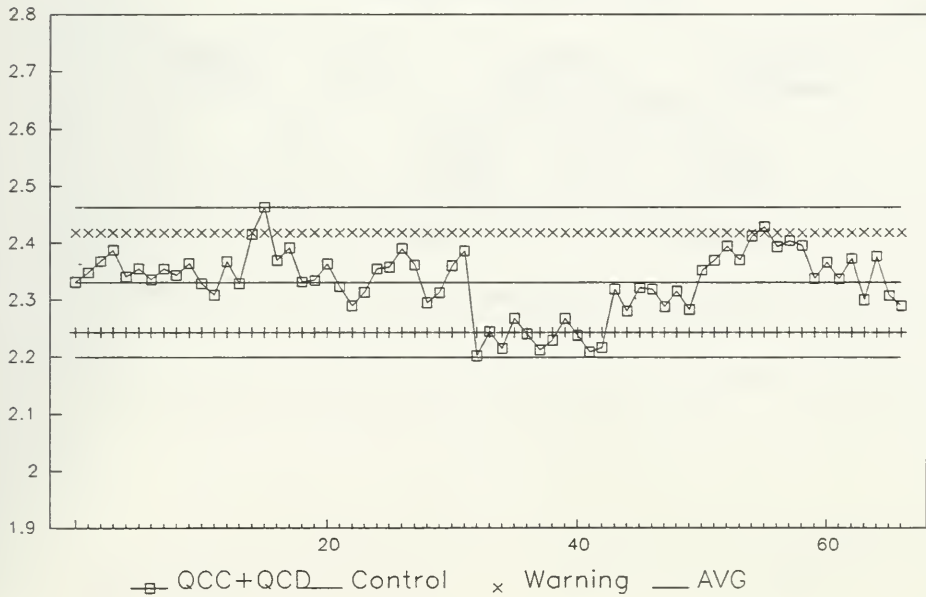
TBMTR — MOLYBDENUM (TOTAL) — MOUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 5 — 20,000 ug/L

Control Samples:

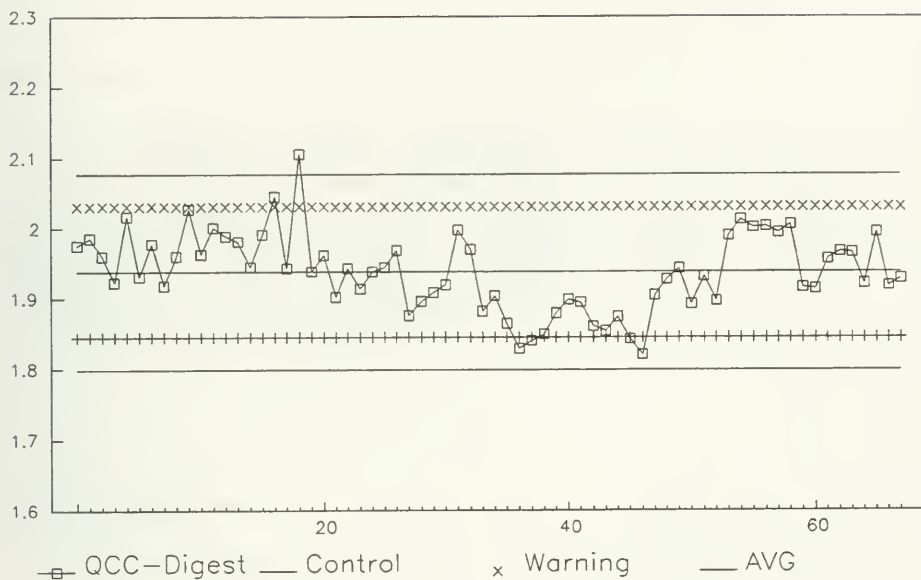|	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.938	96.9	2.9
QCD:	68	0.400	0.385	96.3	3.2
QCC+QCD:	68	2.400	2.323	96.8	2.9
QCC−QCD:	68	1.600	1.553	97.1	3.0

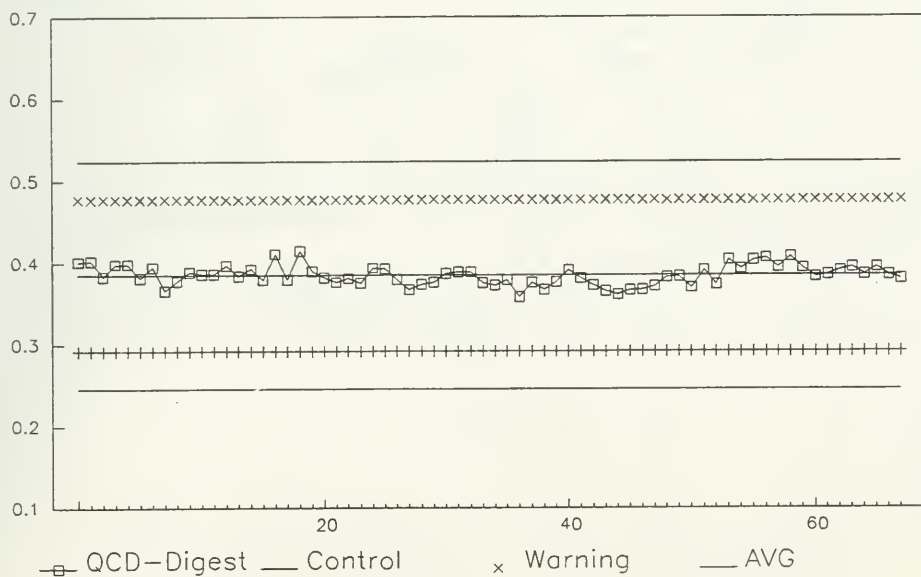For 1994 Control Limits:

$$Sw (C-D) = \quad 0.0463$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
67	5	500	4.6	1.5
N/A	500	5000	—	—
N/A	5000	20000	—	—

Detection Limit (DL) =   5 ug/L

Molybdenum, QCC (TBMTR)

QCC—Digest · Control × Warning · AVG

Molybdenum, QCD (TBMTR)

QCD—Digest · Control × Warning · AVG

Molybdenum, QC Difference (TBMTR)
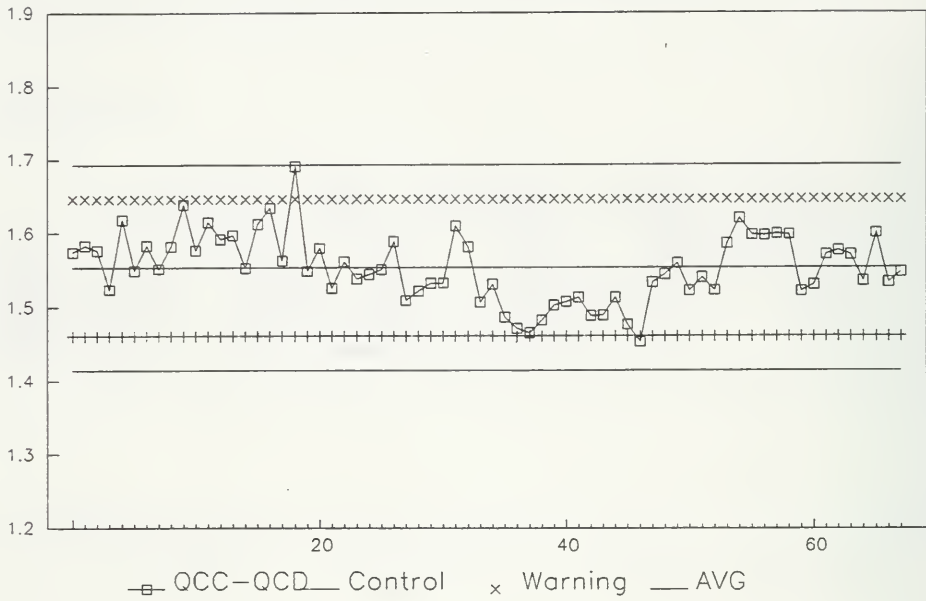
Molybdenum, QC Sum (TBMTR)
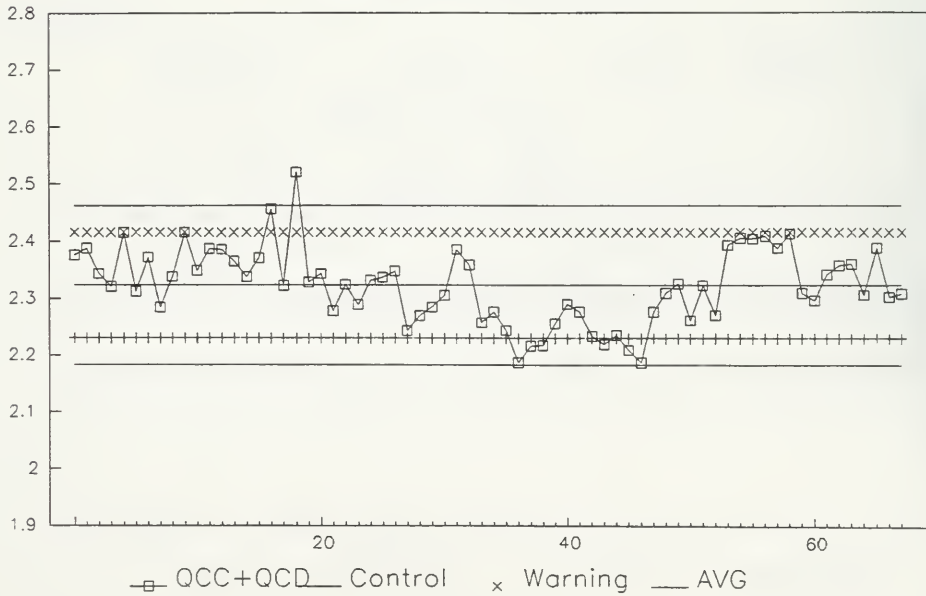
TBMTR – NICKEL (TOTAL) – NIUT

Analytical Range: 5 – 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.926	96.3	3.1
QCD:	68	0.400	0.383	95.8	4.0
QCC+QCD:	68	2.400	2.309	96.2	3.1
QCC−QCD:	68	1.600	1.543	96.4	3.2

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0497$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
63	5	500	46.	2.7
N.A	500	5000	–	–
N.A	5000	20000	–	–

Detection Limit (DL) =   5 ug/L

Nickel, QCC (TBMTR)

QCC–Digest —— Control ×ₓ Warning —— AVG

Nickel, QCD (TBMTR)

QCD–Digest —— Control ×ₓ Warning —— AVG

Nickel, QC Difference (TBMTR)

—□— QCC–QCD __ Control ×Warning __ AVG

Nickel, QC Sum (TBMTR)

—□— QCC+QCD __ Control ×Warning __ AVG

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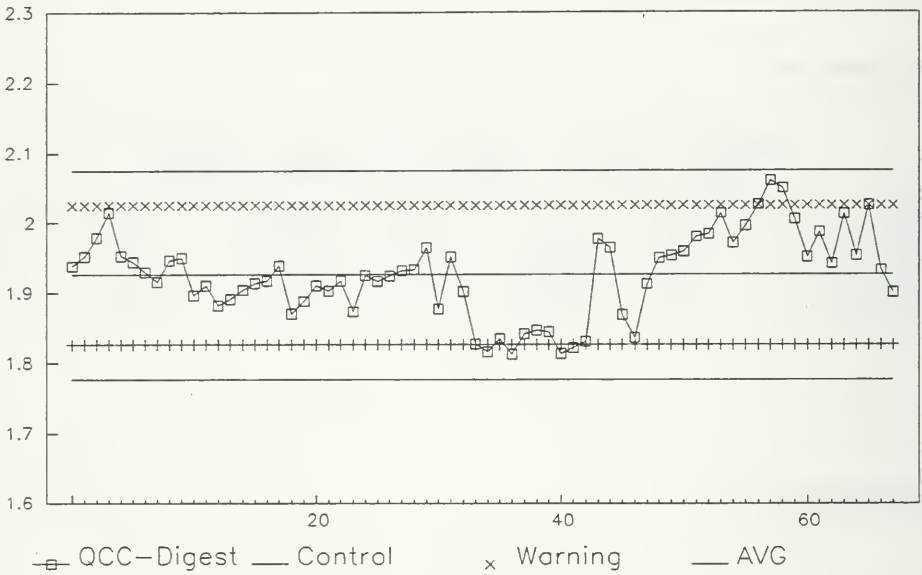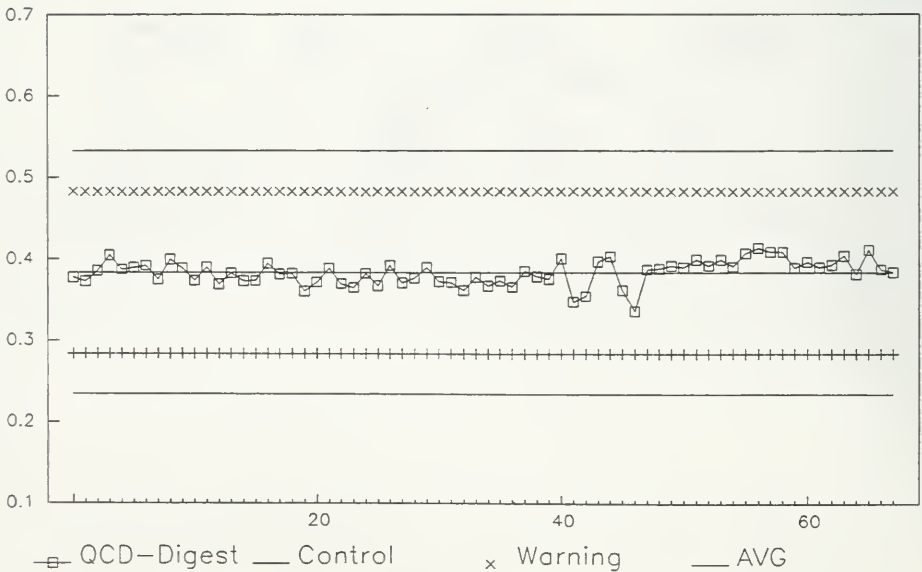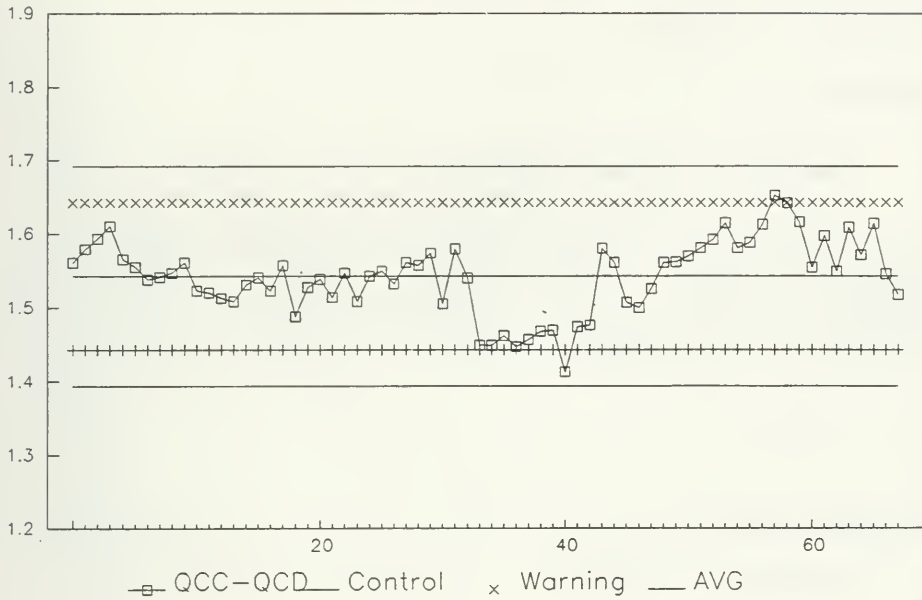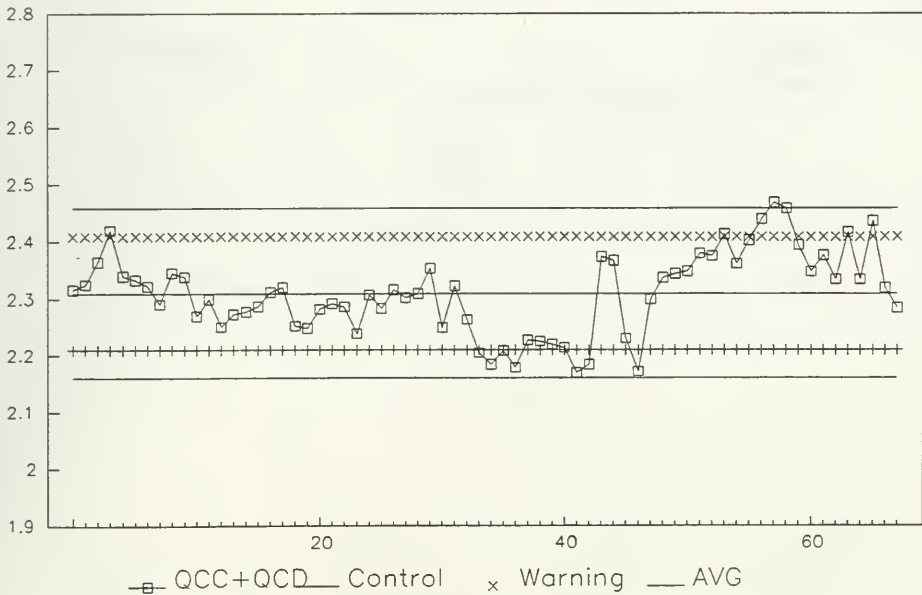
TBMTR — STRONTIUM (TOTAL) — SRUT
Quality Control Data from January 1 to December 31, 1993

Analytical Range: 2 — 10,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	67	1.000	0.981	98.1	2.2
QCD:	67	0.200	0.195	97.5	2.8
QCC+QCD:	67	1.200	1.176	98.0	2.2
QCC−QCD:	67	0.800	0.787	98.4	2.4

For 1994 Control Limits:

$Sw (C-D) =$ 0.0187

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
48	2	200	43	1.2
19	200	2000	560	6.0
N/A	2000	10000	—	—

Detection Limit (DL) =   2 ug/L

Strontium, QCC (TBMTR)
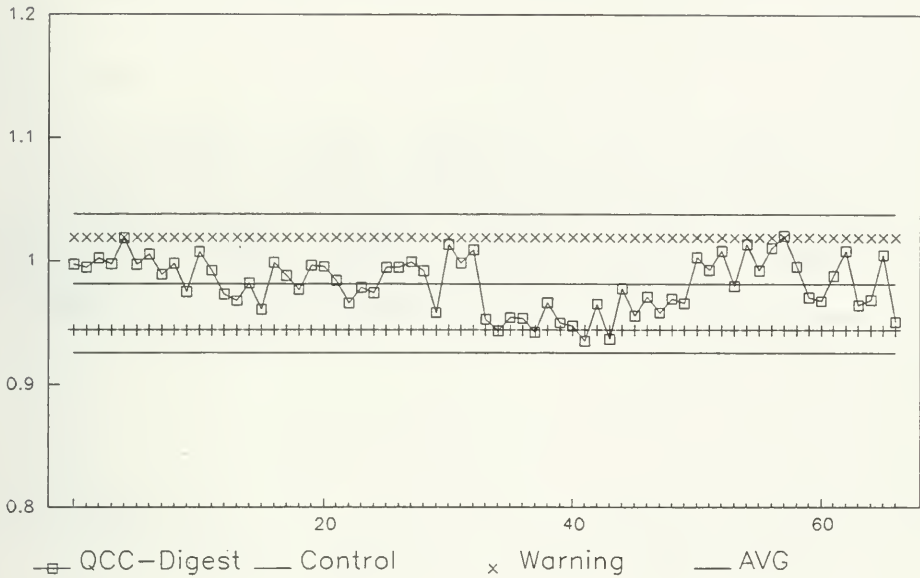
- ▱ QCC–Digest ── Control ── × Warning ── AVG

Strontium, QCD (TBMTR)

- ▱ QCD–Digest ── Control ── × Warning ── AVG

Strontium, QC Difference (TBMTR)

—□— QCC—QCD —— Control   × Warning —— AVG

Strontium, QC Sum (TBMTR)

—□— QCC+QCD —— Control   × Warning —— AVG
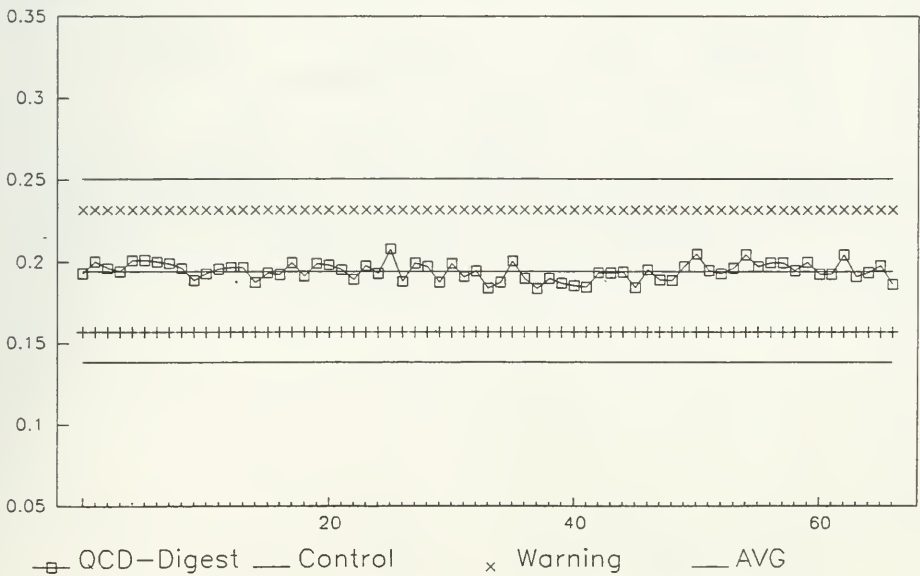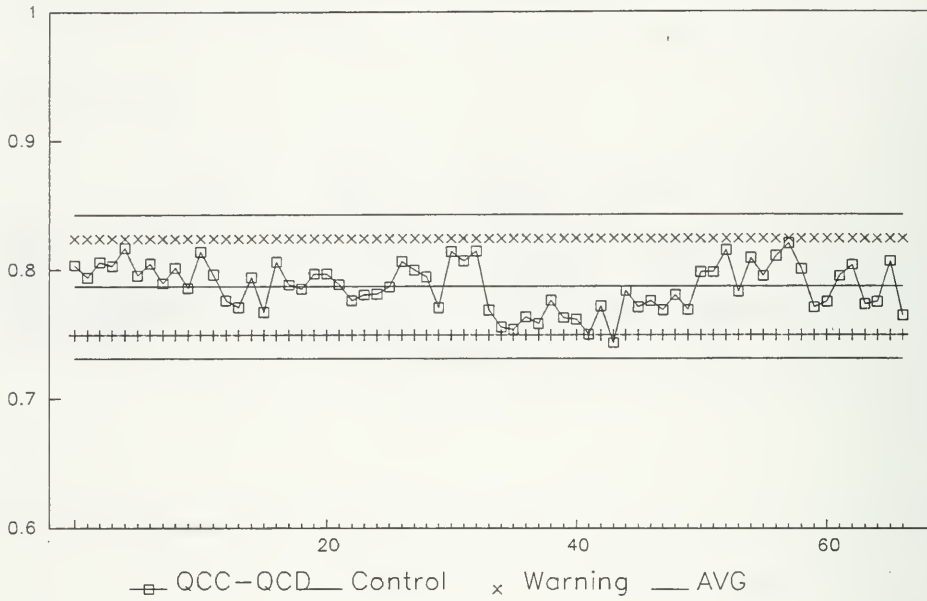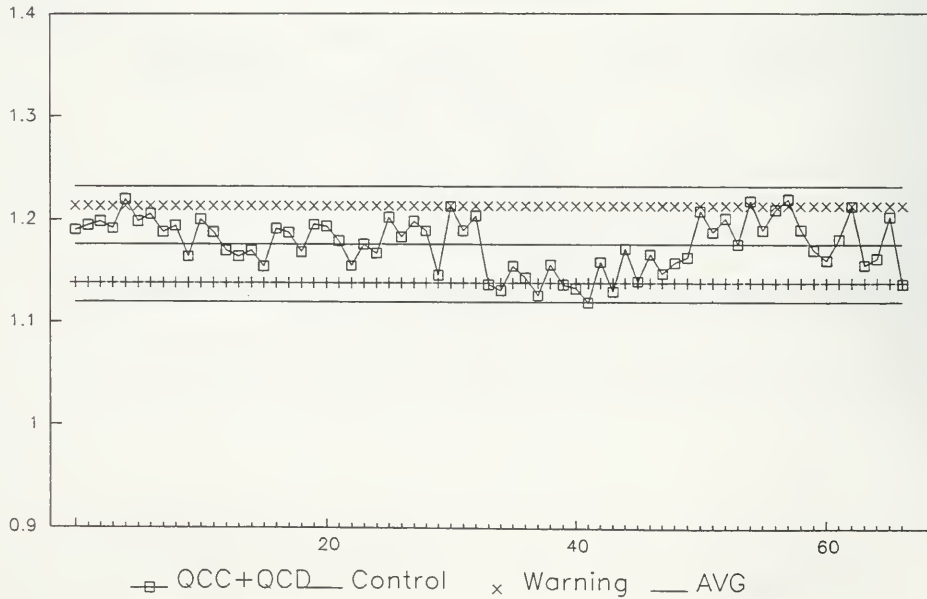
TBMTR — TITANIUM (TOTAL) — TIUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 5 — 15,000 ug/L

Control Samples:

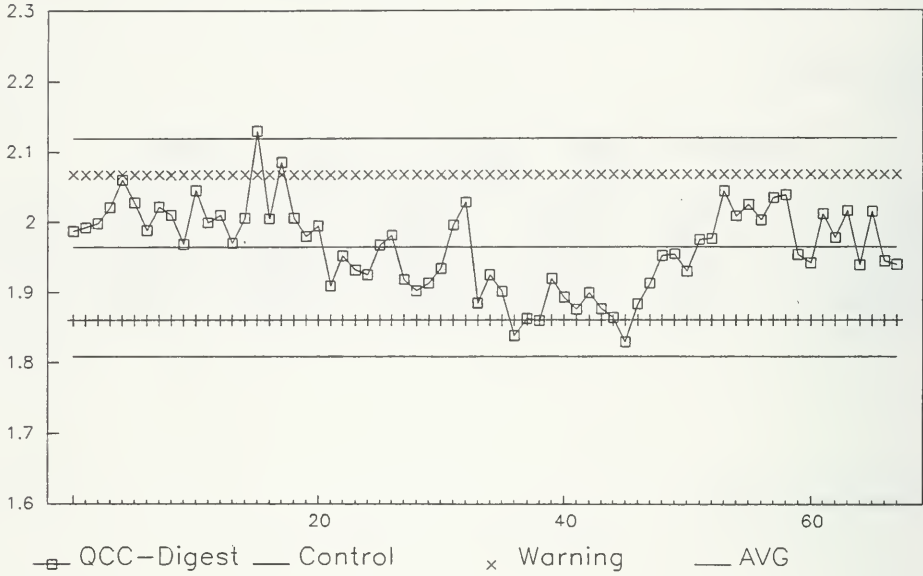|	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.964	98.2	3.1
QCD:	68	0.400	0.386	96.5	3.5
QCC+QCD:	68	2.400	2.350	97.9	3.1
QCC−QCD:	68	1.600	1.578	98.6	3.3

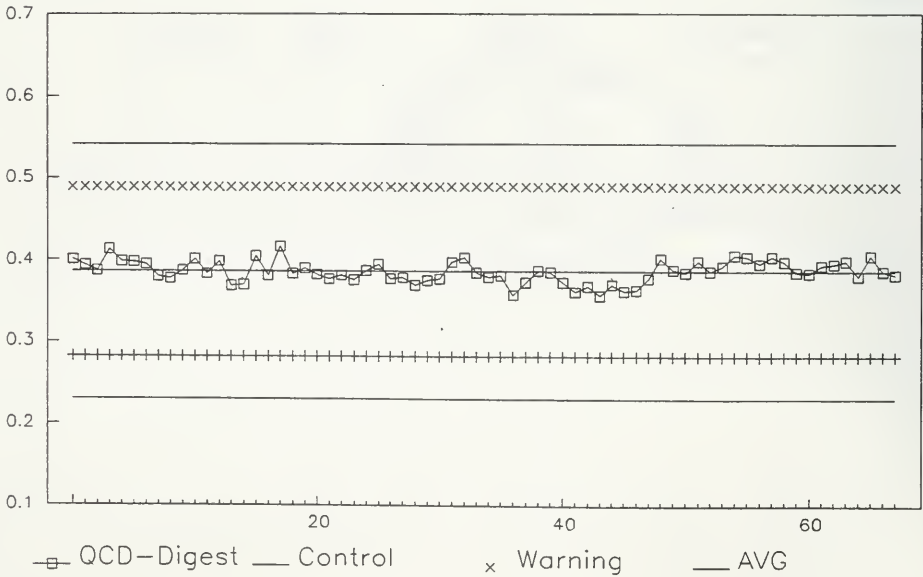For 1994 Control Limits:

$$Sw (C-D) = \quad 0.0518$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
70	5	500	29.	4.4
N/A	500	5000	—	—
N/A	5000	15000	—	—

Detection Limit (DL) = 5 ug/L

Titanium, QCC (TBMTR)

◦ QCC—Digest —— Control ✕ Warning —— AVG

Titanium, QCD (TBMTR)

◦ QCD—Digest —— Control ✕ Warning —— AVG

Titanium, QC Difference (TBMTR)

_◻_ QCC−QCD __ Control   × Warning __ AVG

Titanium, QC Sum (TBMTR)

_◻_ QCC+QCD __ Control   × Warning __ AVG
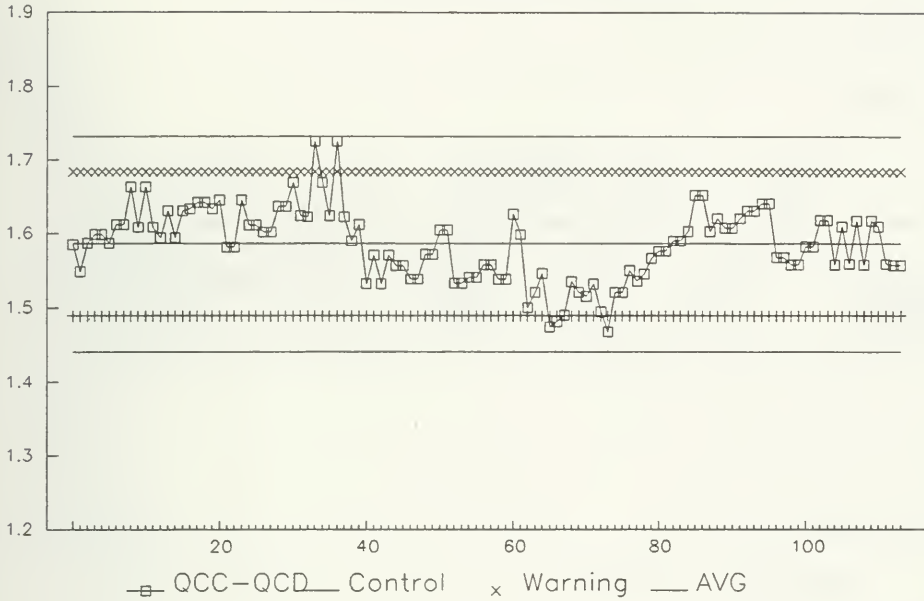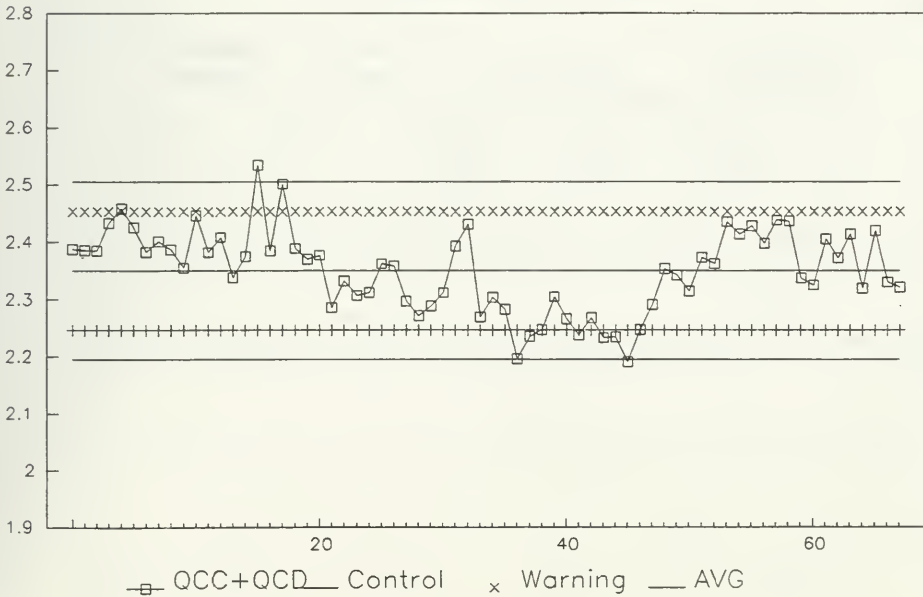
TBMTR — VANADIUM (TOTAL) — VVUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 2 — 25,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.957	97.9	3.0
QCD:	68	0.400	0.386	96.5	3.2
QCC+QCD:	68	2.400	2.343	97.6	3.0
QCC−QCD:	68	1.600	1.570	98.1	3.1

For 1994 Control Limits:

Sw (C−D) =   0.0424

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
69	2	200	5.9	1.3
N/A	200	2000	—	—
N/A	2000	20000	—	—

Detection Limit (DL) = | 2 ug/L |

Vanadium, QCC (TBMTR)

Vanadium, QCD (TBMTR)

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Vanadium, QC Difference (TBMTR)

—□— QCC—QCD —— Control ⨯ Warning —— AVG

Vanadium, QC Sum (TBMTR)

—□— QCC+QCD —— Control ⨯ Warning —— AVG
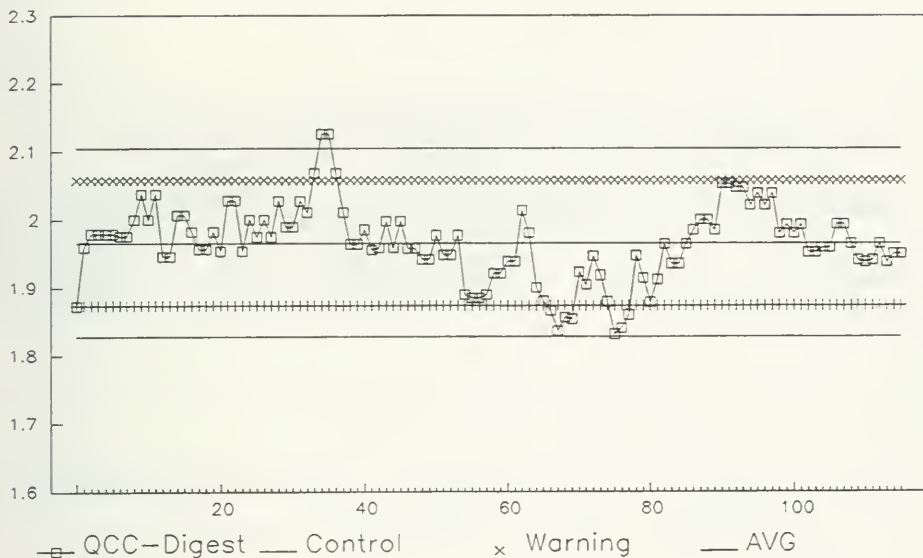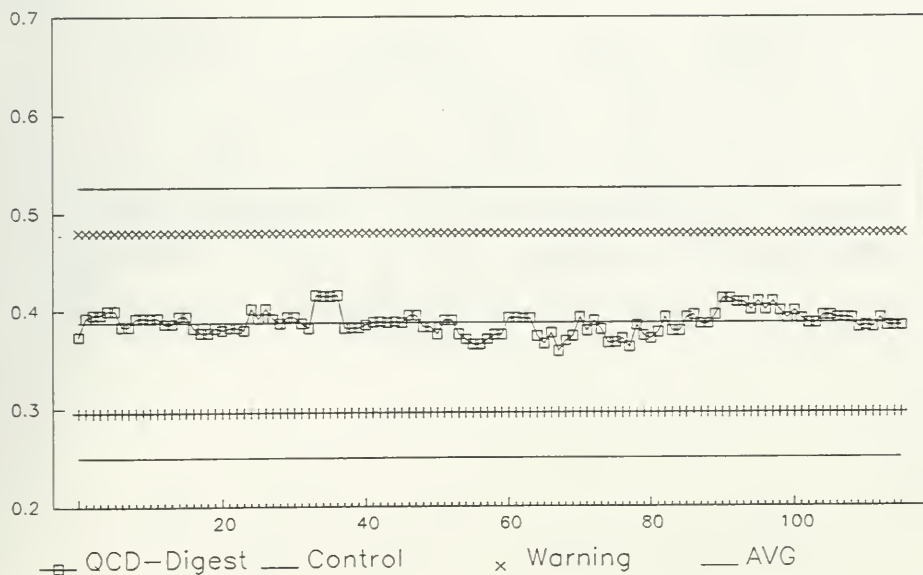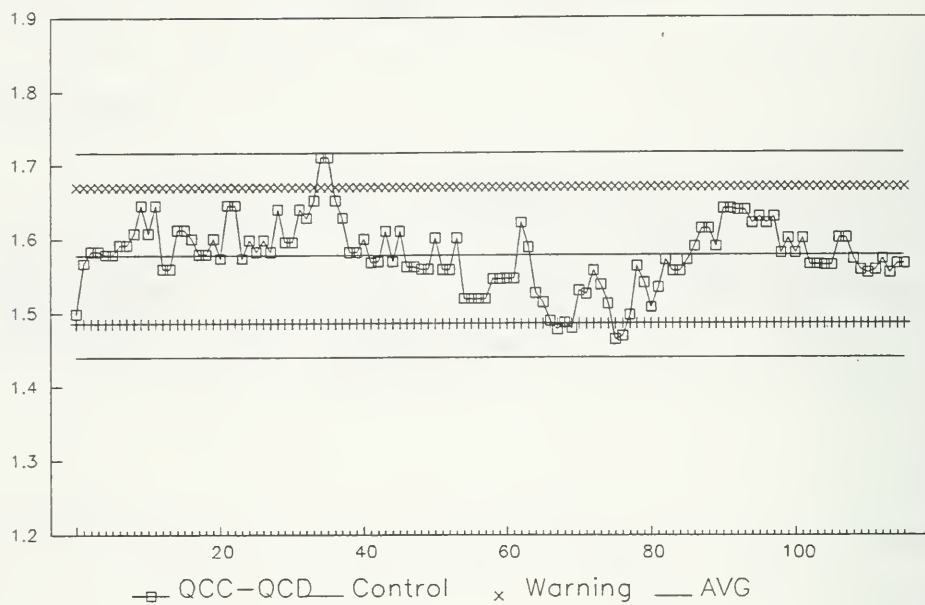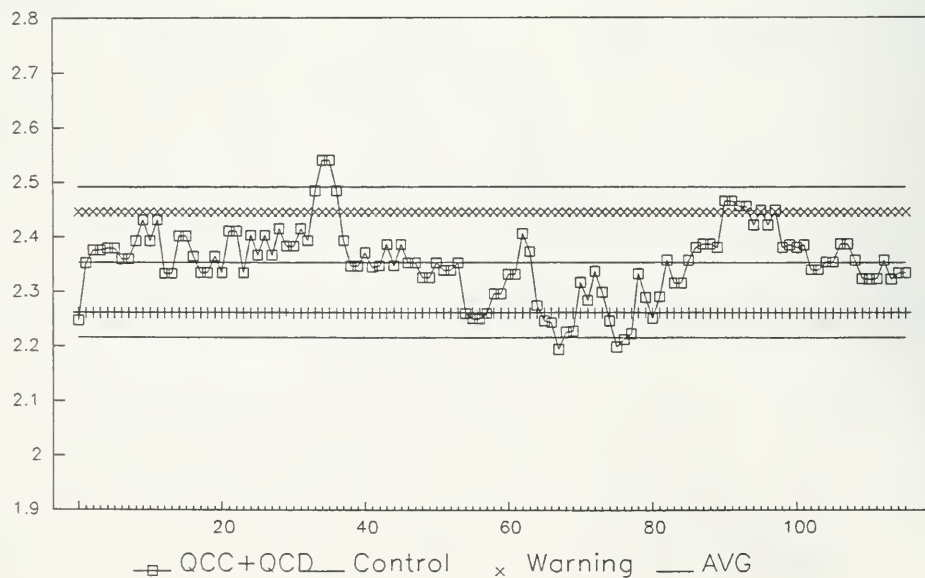
TBMTR – YTTRIUM (TOTAL) – YYUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 1 – 20,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	638	1.000	1.012	101.2	3.1
QCD:	68	0.200	0.196	98.0	2.9
QCC+QCD:	68	1.200	1.207	100.6	3.0
QCC−QCD:	68	0.800	0.816	102.0	3.3

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0269$$

Duplicates:

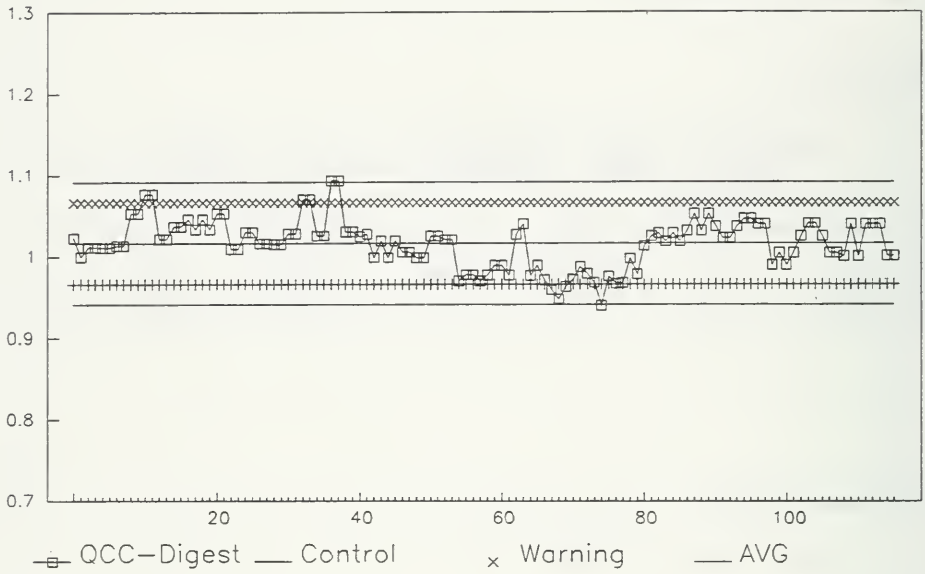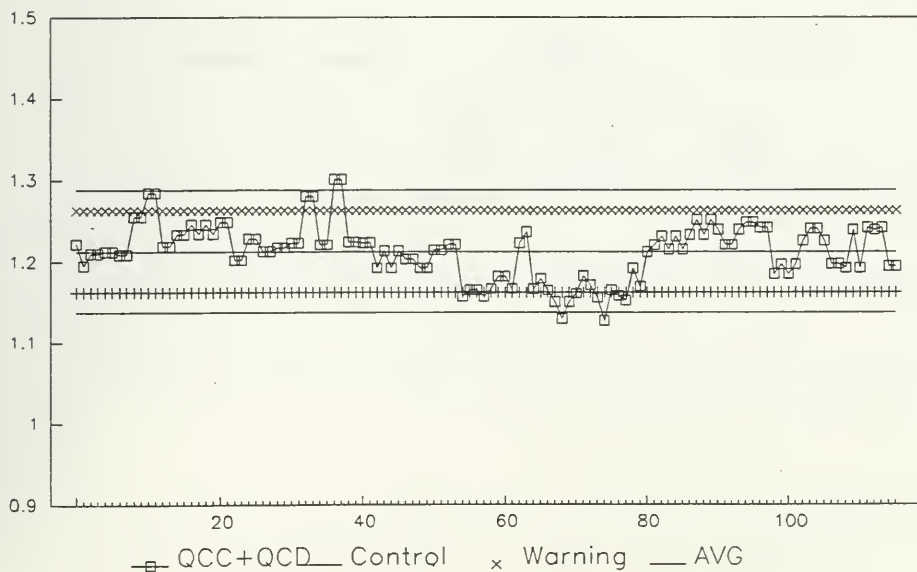| Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
76	1	100	0.94	0.47
N/A	100	1000	–	–
N/A	1000	10000	–	–

Detection Limit (DL) = 　　　1 ug/L

Yttrium, QCC (TBMTR)

QCC–Digest —— Control ✕ Warning —— AVG

Yttrium, QCD (TBMTR)

QCD–Digest —— Control ✕ Warning —— AVG

Yttrium, QC Difference (TBMTR)

QCC−QCD — Control   × Warning — AVG

Yttrium, QC Sum (TBMTR)

QCC+QCD — Control   × Warning — AVG
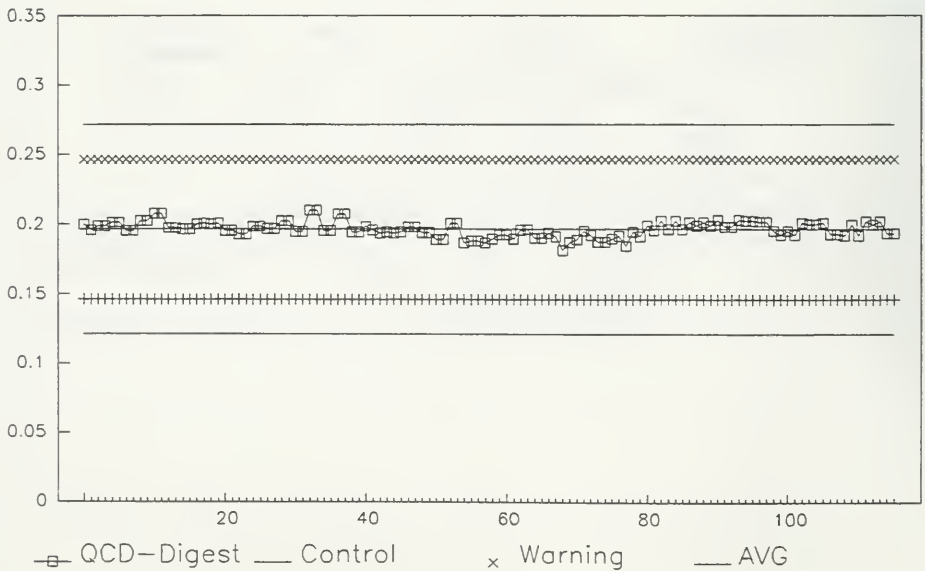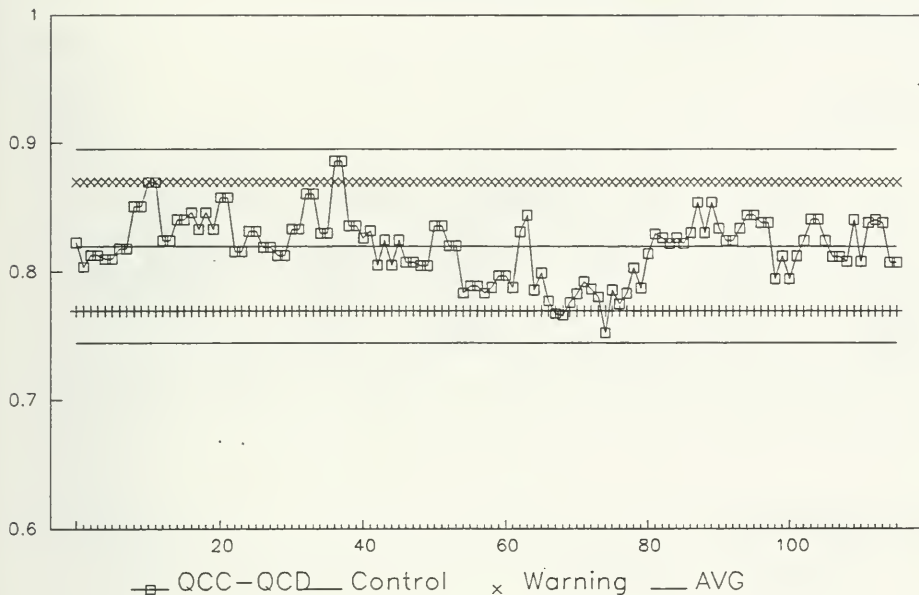
TBMTR – ZINC (TOTAL) – ZNUT

Quality Control Data from January 1 to December 31, 1993

Analytical Range: 10 – 15,000 ug/L

Control Samples:

	Number of Data	Target Conc.	Avg. Conc. Measured	% Recovery	% Rel. Std. Dev.
QCC:	68	2.000	1.912	95.6	3.0
QCD:	68	0.400	0.378	94.5	5.4
QCC+QCD:	68	2.400	2.290	95.4	3.0
QCC−QCD:	68	1.600	1.534	95.9	3.3

For 1994 Control Limits:

$$Sw\,(C-D) = \quad 0.0511$$

Duplicates:

Number of Data Pairs	Sample Conc Span		Mean Value	Standard Deviation
	FROM	TO		
67	10	1000	40.	4.1
N/A	1000	10000	–	–
N/A	10000	15000	–	–

Detection Limit (DL) =   10 ug/L

Zinc, QCC (TBMTR)

□ QCC−Digest ── Control × Warning ── AVG

Zinc, QCD (TBMTR)

□ QCD−Digest ── Control × Warning ── AVG
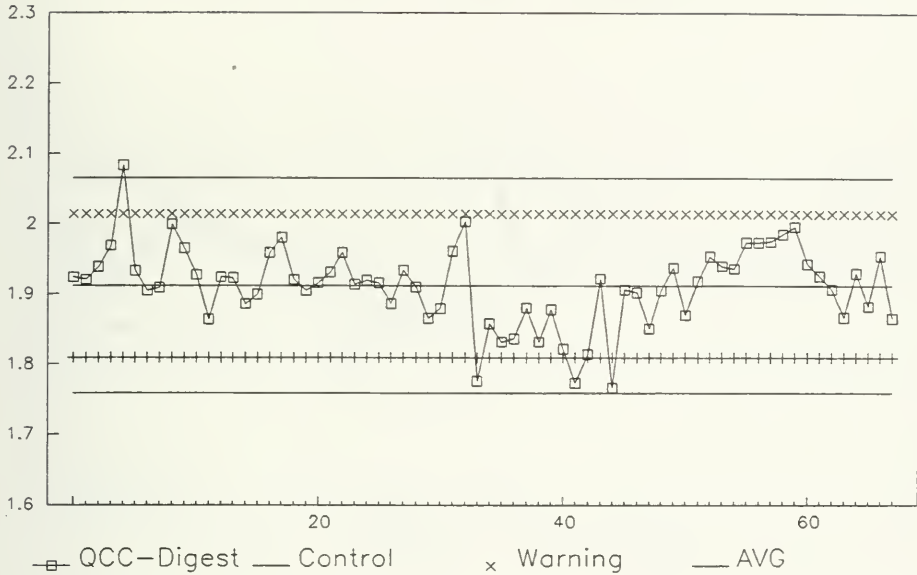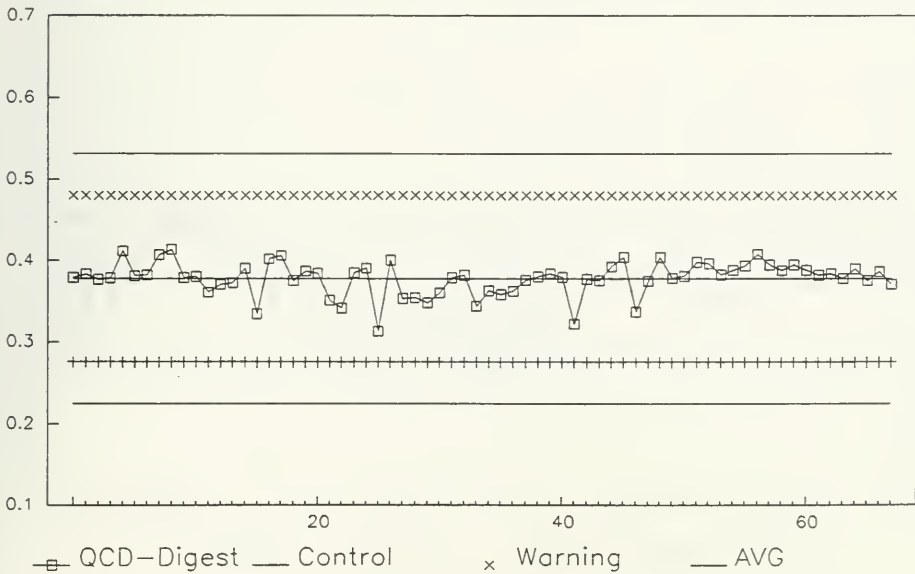
Zinc, QC Difference (TBMTR)

Zinc, QC Sum (TBMTR)